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# Benzo(a) pyrene Content of the Air of American Communities

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Interest in the benzo(a) pyrene content of the atmosphere arises from the facts that it is carcinogenic to experimental animals, is suspected of being carcinogenic to man, and may be a semiquantitative index of the presence in air of other polynuclear hydrocarbons, some of which also are known to be carcinogenic to animals and suspected of producing cancer in man. In addition it has been frequently used as an "index" or measure of carcinogenic potential for comparison with other members of the polynuclear hydrocarbon family. Benzo(a) pyrene, also known as 3,4-benzpyrene (and henceforth referred to in this paper as benzpyrene or BaP), has the structural formula shown in the upper right of Figure 1.

It arises from the distillation or combustion of fuels and is a constituent of tars derived from fuels. Since it melts at 180°C and boils near 500°C, it exists in the atmosphere in the solid phase, usually adsorbed on solid particles which can be recovered from the air by filtration.1 There is evidence that body mechanisms in the lungs may remove this compound from soot, thereby permitting its absorption by cells or body flunds. When these collected particles are extracted with benzene, the benzene-soluble fraction contains benzpyrene. The chromatographic separation of benzpyrene from the benzene extract and the subsequent quantitative analysis have been described in a previous publication.2

This paper reports the results of two series of studies of the benzpyrene content of air. In one, monthly mean values are reported of samples taken continuously for a year (July 1958 through June 1959) in nine United States cities. In the other, a single mean value is reported for each of the 94 urban and 28 nonurban sampling sites for which a pooled sample consisting of about six biweekly 24-hour samples

was collected from January through March, 1959. The latter were regular samples of the National Air Sampling Network, procedures for the collection and analysis of which have been described in previous publications. It is to be noted that the samples herein reported were obtained at a single site at each city. Insofar as possible, the site was in a location judged to be representative of the city's overall atmosphere. However in cities one would expect area to area variation to occur. These same procedures were used to obtain the samples in the nine-city study, except that two or three samplers were run almost continuously throughout the year at one or more sites.

The analytical procedure for benzpyrene does not require large samples. The Network samples were pooled to provide an average. In the nine-city study large samples were taken in order to obtain fractions of benzene extracts sufficiently large to allow experimental study on laboratory animals. These were undertaken at the National Cancer Institute and the University of Southern California Medical School. A report on the results of these studies will be made in the near future.

For analysis, the benzpyrene fraction must be separated from benz(a)anthracene and benzo-(g,h,i) perylene, both of which have ultraviolet absorption bands at 382 m $\mu$  and have in the past sometimes been reported as benzpyrene. Another interference is benzo(k)fluoranthene, which is always found in the benzpyrene fraction. This molecule also absorbs at 382 mu. Therefore its contribution at this wavelength was subtracted to obtain correct values for benzpyrene. The values reported in this paper are believed to be minimal because of the above precautions and because some loss of benzpyrene can occur during collection, extraction, and subsequent handling of samples. The chromatographic recovery of benzpyrene was routinely checked by passing 100-250  $\mu$ g of pure benzpyrene through the alumina column. The recovery ranged about 80%-90%.

Presented at the Twenty-first Annual Meeting of the American Industrial Hygiene Association, Rochester, New York, April, 1960.

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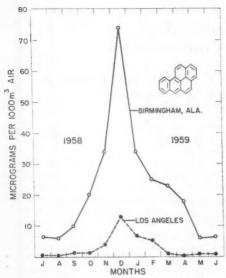


FIGURE 1. One-year study of the monthly change in the concentration of benzpyrene in the air of Los Angeles and Birmingham.

# The Nine-City Study

The study of monthly levels of atmospheric benzpyrene in nine large, widely separated American cities sheds some light on the effects of differences in season, geographic location, and pollutant sources. Benzpyrene concentrations were first computed with respect to the benzene-soluble fraction of airborne particulates (Table I). It will be noted that concentrations were highest in winter in all cities except San Francisco and New Orleans, which had their maxima in the late spring. The minimum in all cases

occurred during the summer. The same trends are, in general, maintained when benzpyrene concentrations are computed with respect to total particulate loadings (Table II), or to air volume (Table III).

The benzpyrene concentration in the airborne particulates, in the benzene-soluble fraction and in the air, is highest for the Birmingham and Detroit sampling sites and lowest for the Los Angeles and San Francisco sites (Figure 1). This geographic variation, possibly associated with fuel use practices and other factors, needs further study.

The amount of particulates in the air is not affected as greatly by the season as is the benzpyrene concentration. In the sampling sites of cities such as Atlanta, Los Angeles, Nashville, and San Francisco, the concentration of airborne particulates reaches a peak in the winter months, although the increase is actually only 11/2 to 4-fold. In the New Orleans and Philadelphia sampling sites, particulate concentration is not as markedly affected by the season. However, in all cases the benzpyrene concentration in the particulates was 3 to 20 times greater during the winter than during the summer (Figures 2 and 3). Comparison of the monthly particulate concentration in the air with the amount of benzpyrene in the particulates indicates a more marked seasonal change in the percentage of benzpyrene than in the amount of airborne particulates.

# The Network Sample Study

The composite samples obtained for the period, January through March, in the 94 urban sites showed a range of 2.4 to 410 µg benzpyrene per gram of particulate, 38 to 2600 µg benzpyrene per gram of benzene-soluble fraction,

Table I

Micrograms Benzpyrene per Gram Benzene-soluble Fraction for Suspended Particulate
Samples Collected July to December, 1958 and January to June, 1959

	1958							1959					
City	July	August	Sep- tember	October	Nov- ember	Dec- ember	January	Feb- ruary	March	April	May	June	
Atlanta	160	340	340	780	830	700	730	690	360	360	-	400	
Birmingham	730	740	1100	1000	1200	1800	1900	1500	1200	1100	710	830	
Cincinnati	390	180	350	960	1200	1200	1200	1500	1900		300	330	
Detroit	950	540	-	1500	1800	1000	1500	2000	1300	950	700	540	
Los Angeles	43	33	55	49	86	230	150	150	74	110	86	49	
Nashville	180		640	1200	1300	1400	1300	erest.		890	380	250	
New Orleans	230	430	350	340	300	340	500	430	310	240	650	400	
Philadelphia	480	300	390	560	850	850	680	400	490	-	480	430	
San Francisco	69	94	140	190	200	260	180	210	180	150	250	280	

TABLE II

Micrograms Benzpyrene per Gram Particulates for Suspended Particulate Samples Collected July to December, 1958 and January to June, 1959

1			19	058		1959						
City	July	August	Sep- tember	October	Nov- ember	Dec- ember	January	Feb- ruary	March	April	May	June
Atlanta	16	36	36	_	99	89	75	65	19	19	-	25
Birmingham	39	38	61	66	125	230	150	125	100	65	35	31
Cincinnati	30	11	31	110	125	120	120	140	180	-	18	20
Detroit	38	21	-	120	125	140	180	280	100	65	41	24
Los Angeles	4.3	2.6	3.9	3.9	11	28	18	18	7.3	13	5.9	4.5
Nashville	11	-	53	150	200	200	160		-	61	26	16
New Orleans	24	44	44	41	40	45	68	48	30	34	64	36
Philadelphia	20	10	25	39	68	68	49	30	39	-	20	21
San Francisco	3.3	6.4	14	-	41	39	23	28	19	11.0	14	16

TABLE III

Micrograms Benzpyrene per 1000 m³ Air for Suspended Particulate Samples Collected July to December, 1958 and January to June, 1959

	1958							1959					
City	July	August	Sep- tember	October	Nov- ember	Dec- ember	January	Feb- ruary	March	April	May	June	
Atlanta	1.6	4.0	4.0	_	15	12	9.9	7.4	2.1	2.1	_	3.6	
Birmingham	6.4	6.1	10	20	34	74	34	25	23	18	5.9	6.3	
Cincinnati	3.9	1.3	2.5	15	14	18	18	18	26	-	2.0	2.1	
Detroit	6.0	4.1	-	18	19	20	28	31	16	12	7.4	3.4	
Los Angeles	.50	.40	1.2	1.2	4.1	13	6.6	5.3	1.1	. 45	.75	.75	
Nashville	1.4	-	6.6	30	55	40	25	-	-	9.0	3.4	2.1	
New Orleans	2.0	4.1	4.1	3.9	3.6	3.9	6.0	4.1	2.6	3.3	5.6	2.6	
Philadelphia	3.5	19	3.6	7.1	12	12	8.8	6.4	6.4	-	3.4	2.5	
San Francisco	.25	.38	1.1	_	3.0	7.5	2.3	2.4	1.3	.75	.75	.88	

and 0.11 to 61  $\mu g$  benzpyrene per 1000 m³ of air (Table IV).

Examination of samples from the 28 nonurban sites gave values from 0.15 to 51  $\mu$ g benzpyrene per gram of particulates, 9.3 to 730  $\mu$ g benzpyrene per gram benzene-soluble fraction, and 0.01 to 1.9  $\mu$ g benzpyrene per 1000 m³ of air (Table V).

Although there is some overlap between urban and rural concentrations, especially for the particulate and benzene-soluble values, the nonurban sites in most cases give lower values. In nonurban areas, the concentration of air particulates is much lower than in urban areas. Consequently the amount of benzpyrene in the air of nonurban communities is much lower than in urban areas. These findings may be graphically demonstrated (Figures 4 and 5) by combining the average January-March, 1959, data from the nine-city study (Tables I and III) with the urban data from the 94 cities in the Network Sample Study (Table IV) and plotting on logarithmic-probability coordinates the data

for the 103 cities on the same figure as that for the 28 nonurban sites (Table V).

The geometric mean values, i.e., 50% values on Figure 4, of benzpyrene concentration in airborne particulates for all U. S. cities and sites measured, are 54 and 8.2  $\mu$ g per gram for the urban and nonurban areas, respectively. The geometric mean values of  $\mu$ g benzpyrene per 1000 m³ of air are 6.6 for urban areas and 0.4 for the nonurban sites (from Figure 5).

When the 103 urban values of µg benzpyrene per 1000 m³ of air are plotted on a map (Figure 6), it will be seen that the country can be divided generally into three areas, the lowest values occurring in the West, and the highest in the eastern and mid-western sections of the country.

Oxidation may in part account for the low concentration of benzpyrene in western cities, especially in California.<sup>5, 6, 7</sup> If so, oxygenated products of benzpyrene should be found in the air, and the ratio of various polynuclear hydrocarbons should vary when determined at the

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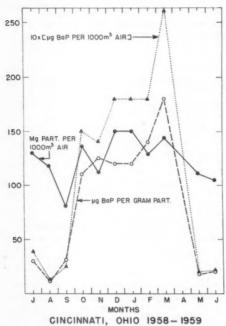


FIGURE 2. Cincinnati, Ohio. One-year study of the monthly changes in: mg particulates per 1000 m³ air; 10× (µg benzpyrene per 1000 m³ air); and µg benzpyrene per 1 gm particulates.

principal source of pollution or in the air. These hypotheses will be examined in the near future, as will the relative rates of emission of benzpyrene from fuel combustion and other sources. The amount of benzpyrene emission data now available is insufficient to explain adequately the range of atmospheric benzpyrene values reported here.

# Comparison of American and Foreign Citieis

The presence of benzpyrene has been demonstrated in the air of several European cities<sup>8-12</sup> (Table VI). In all cases, concentration reaches its maximum in the winter months and its minimum in the summer months. On the basis of the data reported, several of these cities appear to have a greater pollution problem in respect to benzpyrene than any American city included in this study. These determinations were made by methods which may not be entirely comparable to ours. However, they were shown to give values of the same order of magnitude. Our laboratory has analyzed two single samples collected in January and February, 1959, in Lon-

don, England, and found in each 330  $\mu$ g of benzpyrene per gram of particulate.

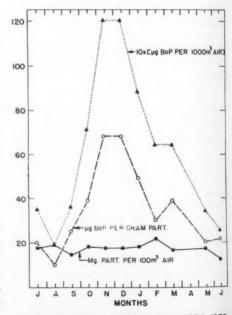
However, the concentration of particulates in the London air is much greater than in most American cities; consequently values of 274 and 366 µg benzpyrene per 1000 m³ of air respectively were obtained for the London samples. These values appear consistent with the earlier determinations in London.

# Human Exposure to Benzpyrene by Inhalation

Interest in the assessment of air concentration of benzpyrene results from its possible effects on an exposed human population. The increase in lung cancer in recent years has been attributed to extrinsic factors, such as soot and fumes of oil and coal furnaces, internal combustion engines, dust from asphalt-surfaced roads and rubber tires, and smoke from eigarettes.

In view of all that is unknown about cancer, there are many variations of opinion as to which extrinsic factor or combination of factors may play the role of prime etiological agent.<sup>7, 13-16</sup>

One means of comparing air pollution and



PHILADELPHIA, PENNSYLVANIA 1958-1959

FIGURE 3. Philadelphia, Pennsylvania. One-year study of the monthly change in: mg particulates per 1000 m<sup>a</sup> air;  $10 \times (\mu g$  benzpyrene per 1000 m<sup>a</sup> air); and  $\mu g$  benzpyrene per 1 gm particulates.

TABLE IV
Benzpyrene Concentrations in Urban Sampling Sites for January through March, 1959.

State	City	μg BaP Per gm Part	pg BaP Per gm Benz. So Fract.	μg BaP Per 1000 m³ Air	State	City	μg BaP Per gm Part	µg BaP Per gm Benz. Sol. Fract.	μg BaP Per 1000 m³ Air
Alabama	Montgomery	340	2000	24	Nevada	Las Vagas	16	160	1.4
Alaska	Anchorage	64	540	3.8	New Hamp-	Manchester	53	600	6.0
Arizona	Phoenix	15	160	5.0	shire				
Arkansas	Little Rock	20	230	1.5	New Jersey	Bayonne	33	410	5.5
California	Berkeley	41	260	2.9		Jersey City	33	440	6.0
Cumina	Glendale	5.3	38	0.8		Newark	46	500	4.5
	San Bernar-	13	140	2.3		Paterson	51	610	6.3
	dino				New Mexico	Albuquerque	15	460	6.3
	San Diego	20	150	2.1	North Carolina	Charlotte	290	2100	39
	San Jose	7.2	91	0.56		Raleigh	180	1300	14
Colorado	Denver	51	290	6.9	North Dakota	Bismarck	5.8	130	0.44
Connecticut	Hartford	68	730	6.5	Ohio	Cleveland	110	1200	24
	New Britain	50	450	5.5		Columbus	70	930	9.5
	New Haven	53	580	5.3		Dayton	78	760	7.9
Delaware	Wilmington	55	650	10		Hamilton	83	600	14
District of	Washington	71	59	9.3		Toledo	100	1200	11
Columbia						Youngstown	190	2000	28
Florida	Miami	28	250	1.9	Oklahoma	Tulsa	13	180	1.0
	Orlando	110	810	11	Oregon	Portland	96	730	8.0
	Tampa	140	1200	15	Pennsylvania	Allentown	26	440	3.4
Georgia	Savannah	>49	>480	>4.3		Altoona*	280	1400	61
Illinois	Chicago	74	950	15		Erie	70	1000	9.5
	Rockford	63	660	7.3		Johnstown	58	660	16
Indiana	East Chicago*	34	710	11.1		Pittsburgh	16	200	5.1
	Indianapolis	120	1100	26		Scranton	33	360	6.1
	Hammond	280	2600	39		York	31	510	5.6
	South Bend	91	200	16	Rhode Island	Providence	24	240	2.9
Iowa	Des Moines	160	1600	23	South Carolina	Charleston	68	530	5.6
Kansas	Topeka	40	510	3.1		Columbia*	120	750	24
	Wichita	19	310	2.3	South Dakota	Sioux Falls	31	480	4.0
Kentucky	Louisville	70	860	16.	Tennessee	Chattanooga	120	1000	31
Louisiana	Shreveport	6.4	150	0.65		Knoxville	210	1900	24
Maine	Portland	180	2100	21	Texas	Beaumont	13	200	0.82
Maryland	Baltimore	64	650	14		Dallas	6.1	160	1.4
Massachusetts	Boston	45	. 730	9.6	1	Galveston	3.4	50	0.16
	Lowell	40	410	3.1		Houston	12	210	1.6
	New Bedford	81	1000	4.4	TT. 1	San Antonio	5.8	110	0.86
Mr. 3.1	Worcester	66	700	14	Utah	Salt Lake City	5.4	54	0.52
Michigan	Dearborn	110	960	9.0	Vermont	Burlington	28	39	1.0
	Flint	140	1400	15	Virginia	Norfolk	59	580	8.4
	Grand Rapids	91	1400	15		Richmond	410	1900	45
Minnesota	Duluth†	110	1500	12	Washington	Roanoke	160	1100	18
	Minneapolis	73	1600	14	Washington Wood Viscinia	Seattle Charleston*	81	790	9.0
Mississippi	Jackson	24	230	1.2	West Virginia	Charleston*	40	900	14
Missouri	Kansas City	46	540	6.5	Wissensin	Wheeling	140	1600	21
	St. Louis*	200	1800	54	Wisconsin	Madison	80	830	4.9
Montana	Helena	2.4	51	0.11	Wavening	Milwaukee	60	730	8.5
Nebraska	Omaha	33	460	3.5	Wyoming Puorto Piero	Cheyenne	36	340	1.2
*1001 DOBA	Omana	99	400	0.0	Puerto Rico	San Juan	15	160	1.2

 $^{\circ}$  In respect to the cities with benzpyrene levels greater than 11  $\mu$ g per 1000 m<sup>3</sup> of air, 5 cities had a concentration of particulates in the air 1.5 to 2 times higher than in the corresponding January to March period in 1958.

†The concentration of particulates in the air for this city in January to March 1959 was ½ that found in the corresponding period of 1958

cigarette smoking has been discussed by Stocks and Campbell. A comparison of the crude estimates of certain pollutants inhaled per year by individuals in diverse environments was found to correlate with lung cancer statistics. In this paper further data representing the possible quantities of benzpyrene that might be inhaled

by humans exposed to the air in various cities and to the smoke of cigarettes are added to help in evaluating this hypothesis (Table VII).

Note must be taken that these data are based on certain speculations and assumptions, including the assumption that the average man inspires approximately 7,000 cubic meters of air a

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Table V
Benzpyrene Concentrations in Non-urban Sampling Sites

State	County	Location	μg BaP Per gm Part	μg BaP Per gm Benz. Sol. Fract.	μg BaP Per 1000 m³ Air
Alabama	Baldwin	Near Ft. Morgan-Gulf State Park	2.9	63	0.076
Arizona	Coconino	Grand Canyon Park	3.0	31	0.041
Arkansas	Montgomery	Quachita National Forest	6.0	79	0.23
California	Humboldt	Trinidad C. G. Station	3.9	100	0.21
Connecticut	Lichtfield	Wigwam Water Reservoir	19	180	0.69
Delaware	Kent	Bombay Hook Wildlife Refuge	8.9	180	0.69
Hawaii	Oahu Island	Near Barbers Point	0.39	11	0.030
Indiana	Montgomery	In Turkey Run State Park	41	430	1.8
Iowa	Clayton	Backbone State Park	9.5	150	0.54
Kentucky	Pulaski	In Cumberland Falls State Park	14	240	0.58
Louisiana	St. Tammany	Near Slidell	4.1	73	0.15
Maine	Hancock	Acadia National Park	4.4	70	0.18
Maryland	Calvert	On Solomon's Island	26	330	0.70
Massachusetts	Nantucket	On Nantucket Island	1.2	25	0.43
Michigan	Huron	Sleeper State Park	40	490	1.2
Minnesota	Cook	Superior National Forest	25	350	0.66
Missouri	Shannon	Missouri St.	0.76	120	0.025
Nebraska	Thomas	Nebraska National Forest	5.4	56	0.10
New Jersey	Cape May	On Cape May	15	230	0.95
North Carolina	Hyde	Near Cape Hatteras	7.9	140	0.25
Ohio	South Bass Island	Perry National Monument	18	400	0.90
Oregon	Curry	Near Cape Blanco	~0.15	~9.3	~0.01
Pennsylvania	Clarion	In Cook Forest	51	730	1.9
Puerto Rico		Loquillo Mountains Park	1.8	23	0.031
Rhode Island	Washington	In Pine Hill Forest	8.3	120	1.9
South Carolina	Richland	Near Pontiac	36	280	1.1
West Virginia	Webster	In Holly Run State Park	33	530	0.89
Wisconsin	Door	Peninsula State Park	25	410	0.72

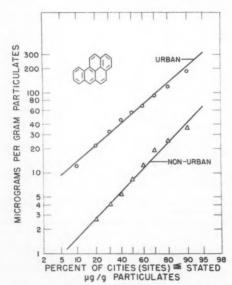


FIGURE 4. Frequency distribution of the benzpyrene concentrations in the airborne particulates of all urban and nonurban sites for January through March, 1959, composite samples.

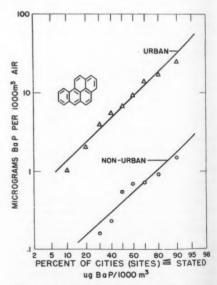


FIGURE 5. Frequency distribution of the benzpyrene concentrations in the air of all urban and nonurban sites for January through March, 1959, composite samples.

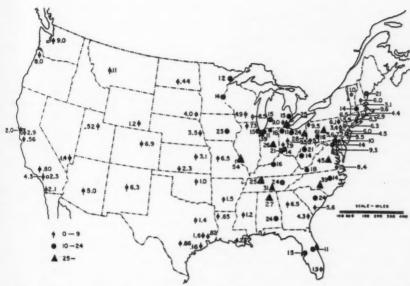


Figure 6. January through March, 1959 concentrations of benzpyrene in  $\mu$ g per 1000 m<sup>3</sup> of air for all urban and nonurban sampling sites as shown on a map of the United States.

Table VI
Variation in Benzpyrene in Various Parts of the World

Month	London, England County Hall*	Milan, Italyt	Sheffield, England*	Cannock, England*	Copenhagen, Denmark‡	Oslo, Norway
January February	147 (1950) 95 "	231 (1958)	78 (1950) 64 "	32 (1950) 27 "	15.4 (1956)	
March	101 "	94 "	65 "	27 "	1	3.45 (1955)
April	48 "	26.6 "	44 "	11 "	1	
May	25 "	3.9 "		16 "	6.2 "	
June	26 (1949)	5.2 44	20 (1949)	4 "	}	0.86 (1955)
July	12 "	3.8 "	24 "	6 (1949)		
August	21 "	2.9 "	21 "	6 "	5.4 "	
September	14 "	11.3 "	33 "	11 "		
October	44 "	38.3 "	58 "	27 44	}	12.4 (1955)
November	80 "	-	56 "	31 "	14.1 "	
December	122 "	_	63	27 "		15.2 (1955)

• Reference 8

† Reference 12

‡ Reference 9

§ Reference 10

year, that the variation in "puffing" and inhaling eigarettes is negligible, that all the benzpyrene inhaled is absorbed or is otherwise effectively retained. Obviously these assumptions are fraught with danger, for it is well substantiated that men performing heavy physical labor inhale more frequently and more deeply, 3e-16 that the size and sex of the individual causes variation in respiration, 3e and that the habits of

individuals in regard to cigarette smoking, driving, and even participation in outdoor activities would have strong influences.<sup>20</sup>

In addition, there is evidence that benzpyrene is not the sole potentially carcinogenic agent present in community air or cigarettes. Benz-(a)anthracene, benzo(b)fluoranthene, benzo(k)-fluoranthene, benzo(g,h,i) perylene and chrysene are known to be present under varying circum-

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TABLE VII

Micrograms of Benzpyrene Inhaled per Year from Ambient Air and from Smoking—Calculated from Mean Annual Concentration

Sampling Sites	μg BaP/Year
Missouri State Forest	0.1
Helena, Montana	~0.8
San Francisco, California	14
Los Angeles, California	20
New Orleans, Louisiana	26
Atlanta, Georgia	44
[Cigarette Smoke (Pack a Day)]	~60
Cincinnati, Ohio	79
Detroit, Michigan	110
Nashville, Tennessee	120
Birmingham, Alabama	150
London (County Hall)*	320

\* Reference 8

stances. However, it is possible that the amount of benzpyrene present may give a crude estimate of the quantity of other polynuclear hydrocarbons inhaled. Other carcinogens which are not polynuclear hydrocarbons may also exist in the air

Finally, there is ample evidence that the absorption or retention of benzpyrene, and possibly other polynuclear hydrocarbons or other carcinogens may be affected by the number, amount and size of particles present in the air, upon which the agents may be absorbed.

However, the information given in Table VII demonstrates that the exposure of an individual to airborne contamination with benzpyrene is lessened as he proceeds to a rural area. In cities of larger size his exposure is increased. This follows the pattern observed in lung cancer mortality rates.

Although massed data do show a correlation between lung cancer mortality rates and community size, study of possible correlation between this 1958–59 benzpyrene data and 1949–51 mortality data on cancer of the lung and other sites (the most recent consistent body of such data available for correlation) has, for individual cities, failed to reveal a significant relationship. A possible explanation is that 1949–51 mortality is presumably associated with exposures to carcinogenic agents in the 1920–50 era which in all probability differ considerably from what is measurable in 1959.

# Discussion

A number of researchers now hold the view that there is no tolerable dose of a carcinogen. Certainly doses as low as  $0.4~\mu g$  of either benz-pyrene or 7.12-dimethylbenz(a)anthyracene

have been shown capable of producing skin tumors in experimental animals. 17, 25, 26 This dose is equivalent to one or two milligrams of urban airborne particulates. Furthermore, numerous observations indicate that carcinogens have a cumulative effect over a period of years. With the tremendous backlog of knowledge on chemically-induced animal cancers and industrial human cancers, the disturbing question arises as to whether many cancers are not derived from constant exposure to an unsafe atmospheric level of carcinogens including benzpyrene especially when endogenous factors are "favorable."

# Summary

Examination of the benzpyrene content in the air of 131 urban and nonurban areas in various parts of the country has disclosed that benzpyrene is universally present. Samples from sites in urban areas yielded higher concentrations of benzpyrene in the air and in airborne particulates than those from nonurban areas. The concentration of benzpyrene at these sites was found to vary from 0.01 to 61 µg per 1000 m3 of air. A 12-month study of the atmospheric benzpyrene concentration in nine large, widely separated American cities has shown that, for the majority, the concentration of benzpyrene in airborne particulates and in the air is at the highest level during the winter months and at the lowest level during the summer months. A map of the different concentrations of benzpyrene found in the air shows a geographic variation which needs a more thorough study. The lower concentration of benzpyrene in the particulates and in the air of California communities may be attributed to less winter heating, use of liquid and gaseous fuels, and in addition may be due to the action of California sunshine and oxidizing atmosphere.

The concentration of benzpyrene in the benzene-soluble fractions of particulates from the air of different urban and nonurban areas varied from 0.00093 to 0.26 per cent. The concentration of benzpyrene in the airborne particulates of urban and nonurban areas varied from 0.00001 to 0.041 per cent. In cigarette tar the concentration of benzpyrene is in the range of 0.00002 to 0.0001 per cent. 21-24

If annual inhalation of benzpyrene may be considered a measure of lung cancer exposure, this exposure is about 100 times greater for an urban resident than a nonurban one, and the exposure is greater for a nonsmoker in many large American cities than for a pack-a-day smoker in a nonurban American community. In many American cities the exposure to benzpyrene of

the urban dweller who smokes a pack a day is double that of the nonsmoking urban resident.

# Acknowledgments

Fulfillment of the undertaking reported in this paper has been due, in large measure, to the many individuals who generously contributed their services. In particular, we mention the many volunteer state and city air pollution personnel all over the country through whose efforts airborne particulate samples were obtained. We also acknowledge the services of Mr. E. C. Tabor and other personnel in the National Air Sampling Network, who extracted the airborne particulate samples to obtain the organic fractions which were necessary for the research work.

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# The Concentrations of Oxidant (Ozone) and Nitrogen Dioxide in the Air of Cincinnati, Ohio

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THE reactions between hydrocarbons and ni-1 trogen dioxide producing "photochemical smog" and oxidant have become increasingly clear in the past few years and are generally accepted as accounting for Los Angeles smog. Interest in "photochemical smog" and the need, for a variety of reasons, for establishing trends in pollution have resulted in several investigations of the occurrence of oxidant and nitrogen dioxide in the atmosphere of a number of communities. Investigations starting in 1954 in Cincinnati and in several other cities in the eastern section of the country were generally of such brief duration or they were made during such times of the year that it was not possible to obtain sufficient data to establish reliably the patterns and trends in the concentrations of oxidant and nitrogen dioxide in the air of these cities.1, 2, 3 For this reason a program for monitoring the air continuously for a variety of pollutants was carried out in Cincinnati over a period extending from January 1, 1957, to December 31, 1959. The trends in the concentrations of sulfur dioxide and particulate matter were described in an earlier publication. The purpose of this paper is to describe the observations concerning the concentrations of oxidant, nitrogen dioxide and nitric oxide.

# Sampling Sites

Three sampling sites were used in this investigation, but the air was monitored only at two stations simultaneously. Site \$1 was in an office-building on a busy commercial street in the downtown business district. This station could be expected to reflect conditions due to heavy motor traffic and the presence of large heating and power plants. Station \$2 was at the Kettering Laboratory in Avondale, about three miles due north of station \$1 and also about two miles east of the Mill Creek Valley, an active industrial area. The station was practically in the center of

local activity, so that it was representative of average, rather than purely industrial or commercial activities. The third station was located in a city park (French Park) about 5.5 miles northeast of the station in Avondale. This site was chosen since it would reflect conditions due to the drift of the pollutants because of south to southwest winds that prevail during the greater part of the year in the Cincinnati area. Air reaching French Park passes over an essentially residential area with a number of thoroughfares carrying heavy motor traffic. Some industry is located in the area between Avondale and the park, but it is generally light in character and cannot be considered a major source of air pollution.

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# **Procedures**

The air was monitored for its content of oxidant, nitrogen dioxide and nitric oxide with automatic equipment fabricated in the laboratory. The equipment followed the general principles and design of the type available commercially and has been described in several publications.<sup>5, 4</sup>

Oxidant was determined by the measurement of the amount of iodine released from a buffered neutral solution of potassium iodide when air was passed through the reagent. The sensitivity of detection of oxidant was of the order of 0.10 parts per hundred million (pphm). A full-scale reading was obtained with 50 parts oxidant per hundred million parts of air. Nitrogen dioxide was determined by measuring the color produced when air was passed through a modified Griess reagent. Nitric oxide was determined separately with the same Griess reagent, following its oxidation to nitrogen dioxide when air was first passed through a weak solution of potassium permanganate.6 One tracing on the chart, therefore, represented nitrogen dioxide per se and, another, the total nitrogen dioxide which included the oxidized nitric oxide. A concentration of 60 pphm of nitrogen dioxide produced a deflection that was twothirds of the full scale of the chart, but as the response of the instrument was logarithmic, there

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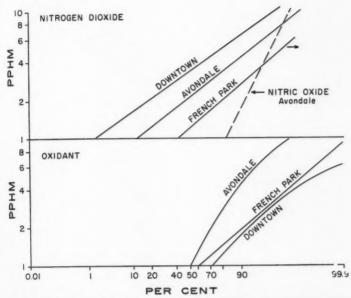


Figure 1. Graphs showing the frequencies of the occurrence of certain concentrations of oxidant and nitrogen dioxide at three stations.

was no difficulty in detecting concentrations of the order of 0.10 pphm or less.

At the start of the program, instruments were available to equip fully only one sampling station, so that after a trial period in January at the Avondale station, the equipment was moved to the downtown station, where it was operated continuously until May, 1958. By June of 1957, duplicate equipment had been built so from that time on air could be monitored simultaneously at two sites. The Avondale station was operated continuously from June, 1957, through December, 1958. In May, 1958, the automatic analyzers were moved from the downtown station to French Park where they were operated during the remainder of the year. In September of 1958, a nitric oxide recorder was completed; it was installed and used at the Avondale station for the remainder of 1958.

During 1959, samples were collected only at the Avondale station, and only during three months of the year; one month in each of the three seasons which we have designated as cold, mild and hot.

# Results and Discussion

# Hourly Concentrations

The frequencies of the occurrence of certain average hourly concentrations of oxidant and

nitrogen dioxide in the air at the three stations and of nitric oxide at the Avondale station are illustrated graphically in Figure 1. As may be seen, higher concentrations of oxidant were found in the Avondale section of the city. Values in excess of 1 pphm occurred 50 per cent of the time at the Avondale station, 38 per cent of the time at the Avondale station, 38 per cent of the time at the perimeter residential station (French Park) and only 26 per cent of the time at the downtown station. An average hourly concentration in excess of 10 pphm was never reached at the downtown station and occurred only 0.01 and 0.05 per cent of the time at the French Park and Avondale stations respectively.

The order of rank of the three stations with respect to the levels of concentration of nitrogen dioxide differed from that shown by the oxidant. The levels of concentration were higher in the downtown area, and decreased in the air at the two residential areas in accordance with their distance from the central core area of the city. Concentrations of nitrogen dioxide, averaging less than 1 pphm per hour were found 44 per cent of the time at the French Park area, 14 per cent of the time at the Avondale station and only 2 per cent of the time at the downtown station.

Data on nitric oxide were collected for several months during 1958 and 1959 only at the Avondale station. These data indicate that the average hourly concentration in Avondale was less

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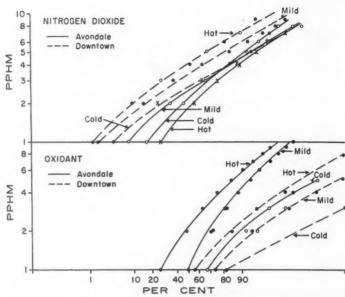


FIGURE 2. Graphs showing the seasonal patterns in the concentrations of oxidant and nitrogen dioxide at two stations.

Table I

Number of Hours of Occurrence of Certain Ranges
of Concentration of Pollutants at
Three Stations in Cincinnati

	Pa	rts per	hundr	ed mill	ion	Total
Station	0-4.9	5.0- 9.9	10.0- 14.9	15.0- 19.9	20.0- 24.9	Total Hours
Oxidant						
Avondale (57-59)	13,769	901	71	11	3	14,755
Downtown (57-58)	9,565	10				9,575
French Park (58)	3,905	20	3			3,928
Nitrogen Dioxide						
Avondale (57-59)	13,637	835	30			14,502
Downtown (57-58)	8,035	1,394	102	10	3	9,544
French Park (58)	4,025	20	1			4,046
Nitric Oxide						
Avondale (58-59)	4,165	70	9	3		4,247

than 1 pphm for 84 per cent of the time. Average hourly concentrations in excess of 10 pphm occurred only 0.5 per cent of the time.

The levels of the concentration of the contaminants in the air at the three stations reflect the sources of the contaminants and the photochemical processes or other reactions involved in the production of oxidant or ozone. Thus, the activities in the downtown commercial area gave rise to high concentrations of nitrogen dioxide and

probably of hydrocarbons, but the concentration of oxidant in the air of the area remained low because the precursor contaminants were moved rapidly out of the area by wind currents, allowing the photochemical process insufficient time to act. By the time the parcel of air had drifted toward Avondale, 3.5 miles north and east of the densely populated and very active basin area, measurable quantities of oxidant were produced by the photochemical process which was accelerated on calm, hot, sunny days. Five and a half miles farther north and eastward in the path of the prevailing summer winds, the sources of pollution were not significant, and the concentrations of the contaminants were reduced by dilution with cleaner air to levels generally lower than those observed at the Avondale station. Shifting winds, however, occasionally moved polluted air toward the French Park area, so that on a few days the atmospheric concentrations of oxidant and nitrogen dioxide at this distant station exceeded those at the station in Avondale.

Seasonal patterns in atmospheric pollution as observed at two stations are indicated by the data plotted in Figure 2. The concentration of oxidant varied seasonally, being at its lowest level at all stations during the cold months (November, December, January and February), increasing gradually to the highest levels during the hot

months (June, July, August and September). The concentrations of nitrogen dioxide do not show this seasonal relationship regularly, and only in the downtown area was such a relation-

ship apparent.

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The number of hours during which the average concentrations of nitrogen dioxide, nitric oxide, and oxidant exceeded certain upper limits are given in Table I. Concentrations of oxidant in excess of 15 pphm per hour were recorded only at the station in Avondale. This time-weighted concentration of oxidant is of interest because it is the standard for ambient air quality, recently adopted by the California State Board of Public Health. The concentration of 15 pphm per hour is the lowest level at which adverse effects were considered to have been observed, and was adopted as the standard after assessing all available data and local experience concerning the occurrence of sensory irritations, damage to vegetation, and reduction in visibility. This does not mean that the same time-weighted average concentration will cause the same effects in other communities. Many factors modify the reactions and extensive observations on the occurrence and effects of the same pollutants in other cities will be needed to establish the validity of this standard, which is likely to be used as a precedent for adoption in other states or metropolitan areas. It was of interest to note that oxidant at a level of 15 pphm per hour or more was found only at the station in Avondale for a total of 14 hours distributed over several days in 1957. On July 18, 1957, concentrations of oxidant in the air ranging from 15.5 pphm to 17.5 pphm occurred for a period of four consecutive hours. The other ten hours during which the average concentration exceeded 15 pphm occurred in August of the same year. An average concentration of 16.3 pphm per hour was present during two consecutive hours on August 8, 1957, and concentrations averaging from 15 pphm to 24 pphm per hour, for two or three consecutive hours each on August 27, 28 and 31. The maximum concentrations of oxidant recorded by the automatic analyzers during each month at the three stations are given in Table II. The highest concentration of oxidant, 29 pphm, occurred at the Avondale station at 10:45 a.m. on August 31, 1957.

Tables III and IV show the peak concentrations of nitrogen dioxide and nitric oxide recorded each month during this investigation. The highest concentration of nitrogen dioxide, 23.7 pphm, was recorded at the downtown station in October of 1957, while a peak concentration of 24.5 pphm nitric oxide was present on November 11, 1958, at the Avondale station.

TABLE II

Maximum Concentration (pphm) of Oxidant During Each Month at Three Stations in Cincinnati, Ohio

	Avondale			Down	French	
	1957	1958	1959	1957	1958	1958
January		6.0			1.5	
February	2.2	7.4		0.3	2.0	
March		4.8		2.3	1.9	
April		6.1		4.0	4.2	
May		13.0	9.0	5.0		4.5
June	10.0	8.3		7.0		5.0
July	17.0	10.4		8.3		2.5
August	29.0	13.5	7.6	9.5		15.5
September	15.0	9.8	1	5.3		3.2
October	8.8	8.8		4.1		8.6
November	6.6	2.4		3.4		6.6
December	5.5	2.2	2.0	1.7		

TABLE III

Maximum Concentration (pphm) of Nitrogen Dioxide During Each Month at Three Stations in Cincinnati

	Avondale			Down	French	
	1957	1958	1959	1957	1958	1958
January		7.0			5.7	
February	7.0	9.0		4.0	6.8	
March		10.0		8.4	7.2	
April		13.0		18.0	17.7	
May		7.5	8.5	19.0		5.0
June		9.3		22.5		6.6
July	11.0	5.5		10.0		8.2
August	17.0	10.3	9.0	16.5		6.5
September	11.5	5.5		14.9		6.7
October	12.3	11.5		23.7		10.7
November	11.6	11.3		17.7		5.7
December	7.0	8.7	8.0	6.1		

TABLE IV

Maximum Concentration (pphm) of Nitric Oxide During Each Month in Avondale

	Avondale		
	1958	1959	
May		6.9	
August		11.8	
September	10.9		
October	10.8		
November	24.5		
December	4.0	22.9	

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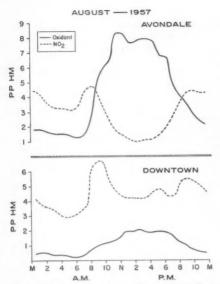


FIGURE 3. Patterns of the average diurnal concentrations of oxidant and nitrogen dioxide at two stations, during August, 1957.

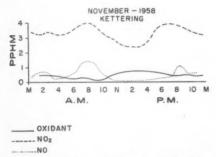


FIGURE 4. Patterns of the average diurnal concentrations of oxidant, nitrogen dioxide and nitric oxide in Avondale (Kettering) during November, 1958.

## Diurnal Patterns

The patterns of the average diurnal concentrations of oxidant and nitrogen dioxide at two stations during August of 1957 are shown in Figure 3. The oxidant was generally at its lowest level at all stations during the night and increased gradually during the day to reach its maximum value between noon and four o'clock in the afternoon. On the other hand, the curves for nitrogen dioxide were often bimodal or even trimodal in character, a peak in the curve being

seen generally between 8 a.m. and 10 a.m., and another during the afternoon or even later in the night. The lowest concentration of nitrogen dioxide was usually found during the time of day when the oxidant was at its highest level.

The pattern of the average diurnal concentrations of nitric oxide in the air at the Avondale station during October, 1958, is shown in Figure 4. Nitric oxide was generally present at low levels of concentration, but the pattern showed the same bimodal character indicated by nitrogen dioxide. Nitric oxide was not always present in the air at Avondale as is indicated by the detailed traces for September 4, 1958, shown in Figure 5. However, on some days as is shown for October 3, 1958, in Figure 5, the traces showed many maxima and minima with the highest concentrations usually occurring in the morning hours and late in the afternoon in parallel with the increase in motor traffic and household activities. In this respect, the patterns of the diurnal concentrations of nitrogen dioxide and nitric oxide were similar to those described by Thomas for Menlo

# Annual Trends

The instrumental continuous monitoring of the air of Cincinnati for its content of oxidant and

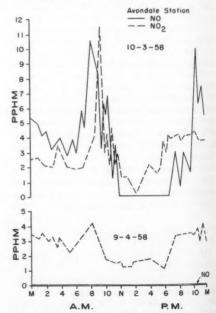


FIGURE 5. Tracings of the fluctuations in the concentrations of nitrogen dioxide and nitric oxide in Avondale on two different days.

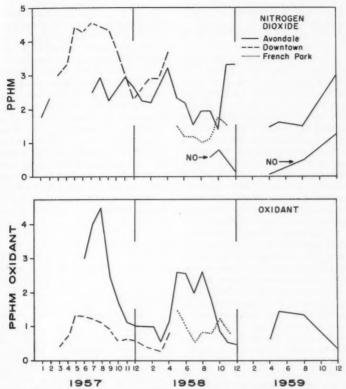


FIGURE 6. Trends in the average monthly concentrations of oxidant, nitrogen dioxide and nitric oxide during 1957-1959.

the oxides of nitrogen was begun as early as 1954. During the period 1954 to 1956, air was monitored for its content of oxidant and nitrogen dioxide only during the months of fall and early winter, periods historically associated with the most frequent occurrence of "smog" in the eastern section of the country. During these months the air generally contained oxidant at very low levels of concentration and the patterns of the fluctuations in concentration were not useful for portraying trends in the concentrations. During 1956, a period of prolonged continuous monitoring for oxidant, extending from April through June, was undertaken at one station. The daily records for this period indicated no concentration of oxidant greater than 5 pphm and an average concentration of only 1.5 parts per hundred million.3 Comparison of the data obtained during the various periods of investigation indicated that the average concentration of oxidant was higher during 1957 than during 1956, 1958 or 1959.

The variations in the average monthly concentrations of nitrogen dioxide and of oxidant for the period 1957-1959 are given in Figure 6. The relationships between the concentrations of oxidant, nitrogen dioxide and nitric oxide and other factors are not shown in the graphs, but an examination of the climatological data for the period indicated that the concentration of oxidant increased with increase in the average monthly temperature. Since hot, sunny days and stagnant conditions favor the photochemical production of oxidant, it is of interest to note that the lowest velocities of wind also occurred during the summer. During the period of this investigation the velocity of the wind during August, for instance, averaged only 5 mph as compared with 7 and 8 mph for the other months of the year. The average monthly temperatures and wind velocities were not significantly different during the three years of this investigation and the decrease in the average concentration of oxidant during 1958 and

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1959 cannot be attributed to marked differences in these climatological factors.

The data for nitrogen dioxide were too few to indicate the trends in the average levels of concentration at all three stations. At the Avondale station where the air was monitored for the longest period of time, no marked differences were observed in the average yearly concentrations during the three years and no relationship could be established between the concentrations and climatologic or seasonal factors. Nitrogen dioxide was always at its highest level at the downtown station, and only at this station was the pattern of the average monthly concentrations similar to that shown by the oxidant.

The photochemical dissociation of nitrogen dioxide into nitric oxide and atomic oxygen is assumed to be the initial step by which solar energy produces "photochemical smog" and oxidants. However, the concentration of nitric oxide does not increase as a result of this dissociation because the nitric oxide formed probably combines with organic matter in the air to produce materials which do not react with the Griess reagent." No relationship could be established between the concentrations of nitrogen dioxide or nitric oxide present at any one time and those of oxidant recorded on any one day.

In the Los Angeles area, the yearly increase in motor car activity is said to be causing an increase of one part per million (ppm) per year in the carbon monoxide content of the air.10 The sampling sites in Cincinnati were not equipped with carbon monoxide recorders, so that the patterns and trends in the concentration of this pollutant could not be established for comparison with those reported for Los Angeles. The intermittent data obtained by the use of a spot sampling technique at each station during the hours when the stations were visited daily, failed to show any increase in the concentration of carbon monoxide. Inasmuch as the analyses for carbon monoxide were generally made at the same hour each day at each station, the data were not indicative of the daily average concentrations or of the fluctuations in the concentrations which could occur during other times of the day. However, the data collected over the three-year period at the Avondale station might be expected to

show a trend, since this station is in the center of local activity. Failure to show a progressive increase in the yearly average concentrations of carbon monoxide, oxidant and nitrogen dioxide. can only mean that so many factors are involved in the accumulation of air pollutants in the atmosphere, that variations in any one factor alone -for example increased activity-cannot be used to predict trends in pollution of the atmosphere. Topography, climatological conditions, technological advances and the size of the expanding community are other factors which must be considered. The shifting of populations and industrial activities to suburban areas surrounding our major metropolitan centers cannot be overlooked, where topography is favorable, as being a major factor affecting the yearly trends in the concentration of air pollutants, particularly toward lower levels.

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# Survey of Air Pollution Research in Europe

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THIS paper is based principally upon information gathered during five weeks of visits in the fall of 1959 to some of the more important groups engaged in or sponsoring air pollution research in England, France, West Germany, Switzerland, and Italy. These visits started with attendance at the International Clean Air Conference in London, and included the headquarters of both the World Health Organization (WHO) in Geneva and the Organization for European Economic Cooperation (OEEC) in Paris. From these latter sources and from the literature, information was obtained on air pollution research in countries not visited.

The Clean Air Conference was held at Seymour Hall in London on October 20-22, 1959, and was sponsored by the (British) National Society for Clean Air. It attracted 1200 attendees, including over 200 from other than Great Britain, representing 29 nations. Seventy-eight<sup>1</sup> papers were presented for discussion by authoritative authors from twelve European countries, Belgium, Czechoslovakia, France, Federal Republic of Germany, Great Britain, Republic of Ireland, Italy, Norway, Poland, Sweden, and the Union of Soviet Socialist Republics, as well as from several non-European nations: Canada, Japan, South Africa, and the United States. Members of the United States delegation were, in addition to the author, John N. vonBergen, representing the (American) Air Pollution Control Association, Ralph C. Graber, Public Health Service, Elmer R. Kaiser, New York University, and Howard P. Willet, Chemical Construction Corporation. There were no Canadian delegates.

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American contributions were papers by the author (with Dr. Harry Heimann as coauthor); Mr. Graber (with Frank Tetzlaff as coauthor); James H. Carter, St. Louis, Missouri; Ellis F. Darley, University of California (Riverside); Arnold E. Reif, Tufts University; Lee Schreibeis, Jr., Allegheny County, Pennsylvania; and Edgar F. Wolf and collaborators, Baltimore Gas and Electric Company. Canadian contributors

were Morris Katz and Kingsley Kay, both of the Department of National Health and Welfare.

The task of identifying the groups carrying on air pollution research in Great Britain was made easy by the recent publication of a list of British air pollution research projects.<sup>2</sup> In Germany, the author was given an equally encyclopedic unpublished list of research projects of the Commission on Air Purification (Kommission Reinhaltung der Luft, Verein Deutscher Ingenieure). The Committee for Applied Research of the European Productivity Agency of the OEEC has a working party on the standardization of methods of air pollution measurement comprising representatives from Austria, Belgium, Federal Republic of Germany, France, Italy, Netherlands, Portugal, United Kingdom, and Sweden. The membership of this working group's is in itself a directory of the agencies in the several nations now engaged in air pollution research. In addition to this working party, the Iron and Steel Committee of OEEC convened a group of European experts on air pollution who were drafting a voluminous report for presentation to that committee early in 1960.

The roster of participants in the Conference on Public Health Aspects of Air Pollution convened by the WHO Regional Committee for Europe in Milan, Italy, in November 1957 was also a Who's Who of European air pollution research. Twenty-one European countries participated, and there were observers from OEEC and the European Coal and Steel Community.

In February 1957, the OEEC published the reports of its Mission No. 145 consisting of representatives of Austria, Belgium, France, Germany, Portugal, Sweden, and the United Kingdom, which visited Belgium, France, Germany, the Netherlands, Sweden, and the United Kingdom during September-November 1953 to study problems of air and water pollution caused by the chemical industry. Both because of the lapse of years between the visits and their reporting, and because the mission's principal interest was in water pollution, this report is less useful than more recent reports in documenting current air pollution research activity in Europe.

Presented at the Twenty-first Annual Meeting of the American Industrial Hygiene Association, Rochester, New York, April 27, 1960.

Information on Soviet air pollution research is based mainly upon the results of a Public Health Service research grant project which has been under way for the last few years for the translation of Russian air pollution literature. The translator, Dr. Benjamin S. Levine, has already published four volumes on studies under way in the USSR and has additional ones in preparation.

# **United Kingdom**

Isaac<sup>2</sup> has classified the air pollution research groups in the United Kingdom into: (a) government departments, (b) government research organizations, (c) nationalized industries, (d) industrial research associations, and (e) universities. The government departments include those of agriculture and forestry, where studies of the effect of SO2 and fluorides on trees and forage crops are under way; the Ministry of Fuel and Power, engaged in studies in Sheffield on smokeless combustion and aerosol behavior; the Meteorological Office, concerned with research on atmospheric turbulence, diffusion and visibility; and the Ministry of Supply, concerned at its Chemical Defense Research Establishment in Porton with both aerosol and meteorological research

The two governmental research organizations active in air pollution research are the Warren Spring Laboratory of the Department of Scientific and Industrial Research and the Medical Research Council. The former is the group responsible for the D.S.I.R. cooperative survey of air pollution, which for many years has been collecting air quality data in the United Kingdom. It is, in adition, concerned with improvement of the methods of measuring air pollutants and of emissions to the atmosphere. The MRC Group for Research on Atmospheric Pollution has its main clinical and research staff at Dunn Laboratories, St. Bartholomew's Hospital in London, but also maintains a working group for study of the epidemiology of respiratory disease at the Department of Social and Industrial Medicine, University of Sheffield in Sheffield. These two MRC groups are the most important ones in Europe, outside of the USSR, working on the health aspects of air pollution.

The main research effort in the United Kingdom is on the reduction of smoke and grit emissions to the atmosphere, and involves work by the National Coal Board, the British Coal Utilization Research Association, and the Department of Fuel Technology of the University of Shefield in the development of smokeless fuels, improved domestic heating appliances, and improved means of grit measurement and control

in industrial furnaces. The Central Electricity Generating Board has placed greatest research emphasis on studies of the behavior of steam power plant plumes, on SO<sub>2</sub>-SO<sub>3</sub> relationships in furnace gases, and on the measurement and dispersion of these gases in the atmosphere. In addition to the above noted nationalized industries, the nationalized atomic energy, gas, and transport industries also undertake research on industry related air pollution problems.

The British Coal Utilization Research Association previously referred to is the most active in air pollution research of a group of industrial research associations, ceramic, coke, internal combustion engine, iron and steel, motor industry, paint, color and varnish manufacture. and steel castings, each of which is devoting some research time to the air pollution problems of its industry. These several research associations maintain their own laboratories and research staffs, and work on problems common to the industry as a whole. In addition to the University of Sheffield, those of London and Durham are engaged in air pollution research studies, the former on the stack plume rise problem, the latter on air pollution measurement methods.

# West Germany

The large and impressive air pollution research program of West Germany is coordinated by a quasi-public commission under the aegis of the German Society of Engineers (VDI). This commission—the Kommission Reinhaltung der Luft—was formed in 1955 with representation from a complete cross-section of German professional, industrial, educational, and governmental associations and interests. It supplements, but does not supplant, the much older VDI Fachgruppe Staubtechnik—Special Committee on Dust Technique. The areas of investigation of these two committees are shown diagrammatically on Figures 1 and 2.

In contrast to the British approach which relies but little on universities, a large part of the German research program is being conducted as university and technical high school research by a system of grants of public funds supplemented by additional funds from industry. The universities and technical high schools thus engaged include those of Braunschweig, Darmstadt, Hamburg, Hannover, Karlsruhe, Munster, and Wurzburg. In addition, work is being done in the laboratories of the Technical Control associations at Koln, Essen, and Berlin, the Hygiene Institutes of Hamburg and Gelsenkirchen, the Clean Air Institute Station, Essen, and the Dust Research Institute, Bonn. The areas of greatest support have to do with SO<sub>2</sub> and its life

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cycle—formation, emission, transport, diffusion and effect on receptors. Of these, the studies of effect of SO<sub>2</sub> on vegetation at Biersdorf have been the most extensive.

As is evident from the large number of industries and sources listed in Figure 2 under the heading, "Origin and emission of dusts and gases," studies on the nature of industrial emis-

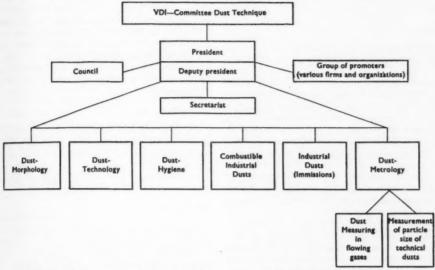


Figure 1. Organization of VDI Special Committee on Dust Technique. (Reproduced with permission from Journal of the Institute of Fuel.) $^{9}$ 

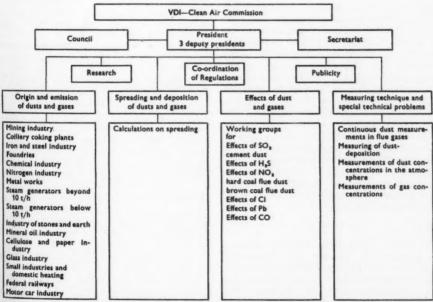


FIGURE 2. Organization of VDI Clean Air Commission. (Reproduced with permission from Journal of the Institute of Fuel.)<sup>a</sup>

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sions and their control represent an important segment of the German program. One result of these studies is a series of VDI standards for industrial dust emission which have been incorporated in the VDI Handbook Reinhaltung der Luft.<sup>7</sup>

# France

French air pollution research is centered almost entirely in Paris, with greatest emphasis on measurement of the quality of the air of Paris by the personnel of the Hygiene Laboratory of the City of Paris. Carbon dioxide, sulfur dioxide and dust are measured at a network of about a dozen stations, one of which measures at three elevations on the second highest structure in Paris, the Tower St. Jacques. In France, neither the British pattern of industrial research associations and research in nationalized industries, nor the German pattern of sponsored university research is followed. French research on control equipment and methods is pursued by equipment manufacturers or users.

Although there exists in France both an Interministerial Commission for the Study of Air Pollution and an Association for the Prevention of Air Pollution, there is no indication that either sponsors or conducts research.

## Italy

Just as French air pollution research is centered in Paris, so Italian air pollution studies are centered in Milan, but in this case, about a complex of university, provincial, and national laboratories—the Institute of Hygiene of Milan University, the Chemical Laboratory of the Province of Milan, and the Fuel Experiment Station of the Italian Ministry of Industry and Commerce. The studies in the Milan Laboratories are intensive, and cover in addition to the more common studies of CO2, SO2, and particulate matter in the air of Milan, investigations of lead and polynuclear hydrocarbons. There is great local interest in the latter, particularly in benzpyrene, and some quite elegant studies in this regard are under way. Outside of Milan, air quality measurements have been made in Genoa, Mestre, Padua, and Rome.

# Belgium

The Institute for the Encouragement of Scientific Research in Industry and Agriculture (IRSIA) has, by grants, supported air pollution studies at the Botanical Institute of Liege, the State Agronomical Institute (Gemblaux), the University of Ghent, the Royal Meteorological Institute, and the Institutes of Industrial Chem-

istry and of Pathology at Liege University. These studies cover air pollution effects upon vegetation and upon man, measurements of air quality and research on meteorological and climatological aspects of air pollution.

The Town Planning Department of the Ministry of Public Works has included air pollution as one of the factors under study as part of a general planning study of the Liege region. The investigations have been subdivided into chemical, meteorological, vegetation and health effects studies, as well as a detailed source inventory of the region.

# The Netherlands

The technical high schools of the Netherlands have been the principal centers at which air pollution studies have been made. These have included studies of industrial dust collectors. An improved form of strip filter paper sampler is under development in the Netherlands.

# U.S.S.R.

Soviet air pollution research is coordinated by the Committee on the Determination of Limits of Allowable Concentrations of Harmful Substances in Atmospheric Air and the Norms for Discharges into the Atmosphere, which committee is affiliated with the Main State Sanitary Inspectorate of the USSR Ministry of Health. The headquarters of the committee and its work is the F. F. Erisman State Scientific Research Sanitary Institute, Moscow. However, on the twenty-six member committee are representatives of about eighteen different research institutes, most of which have one or more air pollution research groups. These institutes are mainly in Moscow, Leningrad, and Kiev. In Moscow are the Central Institute for Advanced Training of Physicians, Institute of General and Communal Hygiene, Institute of Industrial Hygiene and Occupational Diseases, Moscow Medical Institute, Moscow Regional Sanitary Hygiene Institute, Sanitary Epidemiological Station of the City of Moscow, and the above noted F. F. Erisman Institute. In Leningrad are the Leningrad Medical Institute for Sanitation and Hygiene, Leningrad Scientific Research Institute of Industrial Hygiene and Occupational Diseases, and Leningrad Institute of Radiation Hygiene. In Kiev are the Ukrainian Scientific Research Institute of Municipal Hygiene and the Ukrainian Institute of Communal Hygiene.

All the above are medical institutes, and as such, are primarily concerned with clinical research involving humans and experimental studies involving animals. They also undertake studies involving animals.

ies of methodology of air pollution measurement, and apply these methods to air quality measurements. The general research approach of the medical institutes has been the assignment of a specific pollutant (e.g., H<sub>2</sub>S) to an institute. The institute is expected to study all aspects of the pollutant, its sources, concentration in the air, and its effects, and to recommend a safe limit for emission of and exposure to the pollutant.

One unique aspect of Soviet research to set safe limits for pollutants has been the use of Pavlovian techniques of aberrations in conditioned reflexes caused by exposure of animals to pollutants. By these and other techniques, the Russians have compiled a list of thirty substances for which limits for exposure to maximum concentration at one time and for a twenty-four hour average exposure have been set.<sup>1</sup>

The design and construction of air pollution control equipment is the responsibility of the trust "Gazo-otchistka".

# Czechoslovakia

Air quality measurements of dustfall,  $SO_2$ ,  $Cl_2$ , and  $NO_x$  are being made in every town district in Czechoslovakia by laboratories of several research institutes and by the Academy of Sciences. These institutes and the Academy are working on problems of air cleaning and of damage to vegetation, particularly spruce, pine, and hops. The Soviet air quality standards are used in Czechoslovakia.

# Other Countries of Europe

Austria, the Netherlands, Portugal, and Sweden are participants in the OEEC working party for Standardization of Methods of Measuring Air Pollution. APCA Abstracts' for the past two years list papers from authors in all these countries except Portugal, and in addition, from Denmark, East Germany, Hungary, Poland, and Switzerland. There must therefore be air pollution research activity in all these lands. However, the information at hand is insufficient to discuss the work in any detail.

## Conclusion

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The air of European metropolises tends to be more visibly polluted than that of comparable American cities, a difference most likely attributable to the widespread availability of natural gas in America. This has pointed the need for both air pollution research and control in each of the European nations. The total air pollution research effort of Europe is comparable in size and scope to that in America. In America, the

results of work in all states and provinces are rapidly disseminated at frequent national conferences and seminars, whereas in Europe, interchange of information is impeded by language and political barriers that tend to dilute the large total effort by requiring each nation to duplicate much of the same work.

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# Appendix

Addresses of European Organizations Engaged in Air Pollution Research

## BELGIUM

- L'Administration de l'Hygiene et de la Medecine du Travail Ministere du Travail et de Prevoyance Sociale
- 2, rue Lambermont, Bruxelles
- University of Liege
- 2, rue Armand Stevart, Liege

## ENGLAND

- Meteorological Office, Air Ministry Kingsway, London, W.C. 2
- Research and Development Department Central Electricity Generating Board
- Friars House, Blackfriars Road, London, S.E. 1 British Iron and Steel Research Association 140 Battersea Park Road, London, S.W. 11
- British Coke Research Association 74. Grosvenor Street. London, W.1

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Research Department London Transport Executive 55, Broadway, Westminster, London, S.W. 1

National Society for Clean Air Palace Chambers, Bridge Street, London, S.W. 1 Group for Research on Atmospheric Pollution

Medical Research Council Dunn Laboratories, St. Bartholomew's Hospital West Smithfield, London, E.C. 1

National Agricultural Advisory Service Ministry of Agriculture, Fisheries & Food Great Westminister House, Horseferry Road, London, S. W. 1

Imperial College of Science and Technology University of London Exhibition Road, South Kensington, London, S.W. 7

British Non-Ferrous Metals Research Association 81 Eviston St., London, N.W 1

Department of Fuel Technology and Chemical Engineering
University of Sheffield

Mappin Street (St. George's Square), Sheffield, 1 British Steel Castings Research Association

East Bank Road, Sheffield, 2 Safety in Mines Research Establishment

Ministry of Fuel and Power Portobello Street, Sheffield, 1

Coal Research Establishment National Coal Board Cheltenham, Stoke Orchard, Gloucestershire

Paint Research Station Research Association of British Paint Colour & Varnish Manufacturers

Waldgrave Road, Teddington, Middlesex British Ceramic Research Association Queens Road, Penkhull, Stoke on Trent

Central Veterinary Laboratory Ministry of Agriculture, Fisheries & Food New Haw, Weybridge, Surrey

Public Health Engineering Section
Department of Civil Engineering, University of Durham
King's College, Newcastle upon Tyne, 2

British Internal Combustion Engine Research Association 111-112, Buckingham Avenue, Slough, Buckinghamshire

The British Coal Utilization Research Association Randalls Road, Leatherhead, Surrey

Industrial Group, Windscale Works United Kingdom Atomic Energy Authority Sellafield, Calderbridge, Cumberland

Chemical Defense Experimental Establishment Ministry of Supply Porton Downs, Salisbury, Wiltshire

British Motor Industry Research Association Lindley, Nr. Nuneaton, Warwickshire

Atmospheric Pollution Division, Warren Spring Laboratory

Department of Scientific and Industrial Research Stevenage, Hertfordshire

National Chemical and Physical Laboratories Teddington, Middlesex

Atomic Energy Research Establishment Didcot, Harwell, Berkshire Forestry Commission 25 Saville Road, London, W. 1.

#### FRANCE

Applied Research Division European Productivity Agency Organization for European Economic Cooperation 3 Rue Andre-Pascal Paris-XVIe

Service du Laboratoire d'Hygiene de la Ville de Paris 1 bis, rue des Hospitalieres, St. Gervais, Paris IV

La Commission Interministerielle de l'Etude de la Pollution Atmospherique Ministere de la Sante, 11 rue de Tilsit, Paris

Laboratoire Central des Services Chimiques de l'Etat-12 Quai Henri IV, Paris

#### GERMANY

Institute fur Wasser, Boden und Luft Hygiene Conensplatz 1, Berlin-Dahlen

Kommission Reinhaltung der Luft Verein Deutscher Ingenieure Prinz-Georg-Strasse 77/79 Dusseldorf 10

Hygiene-Institut der Hansesstadt Hamburg Gorch-Fock Wall 15 Hamburg 36

Forschungsinstitut für Luftreinhaltung e. V. Alfred-Pott-Weg 8 Essen-Bredeney

Staubforschungs-Institut des Hauptverbandes ger gewerblichen Berufsgenossenschaften e V. Longwartweg, 103, Bonn

Fachgruppe Staubtechnik Verein Deutscher Ingenieure Prinz-Georg-Strasse 77/79, Dusseldorf 10

Technische Hochschule Carolo Wilhelmina Pochelstrasse 4, Braunschweig

Technische Hochschule Hochschulstrasse, Darmstadt

University of Hamburg Edmun-siemers-alle, Hamburg

Technische Hochschule Am Weisengarten 1. Hannover

Technische Hochschule Fridericiana Kaiserstrasse 12, Karlsruhe

University of Munster Chlossplatz 2, Munster

Julius Maximilians University Sanderring 2, Wurzburg

Technischer Uberwachungs-Verein e.V. Steubenstrasse 53 Essen

Farbwerke Hoechst A. G. Anorganische Abteilung Frankfort n. M. -Hoechst

Landesanstalt fur Bodennutzungsschutz des Landes Nordheim-Westfalen Bochum

Staatsinstitut fur Angewandte Botanik Auden Kirchhofen Hamburg Institut fur Tierernahrungslehre Der Landwirtschaftlichen Hochschule Stuttgart-Hohenheim

#### PALY

Laboratorio Chimico Provincia di Milan Milan

Stazione Sperimentale per i Combustibili Politecnico di Milano San Donato Milanese

Monte Cimone Observatory Sestala, Modena

# SWITZERLAND

Division of Environmental Sanitation World Health Organization Palais des Nations, Geneva

#### THE NETHERLANDS

Afdeling Gezondheidstechniek T.N.O. Apelfoornselaan 149, 'n-Gravenhage Leberatorium voor Warnita en Stoftech

Laboratorium voor Warnite en Stoftechnich Technische Hageschool, Delft

Institut Voor Plantens ziektenkundig Onderzoek Binnenhaven 4 Wageningen

UNION OF SOVIET SOCIALIST REPUBLICS

Azerbaidzhan Medical Institute Ul. Karganova, 11/13 Baku, Az SSR Gor'kii Institute of Epidemiology and Microbiology Gor'kii, RSFSR

Ukrainian Scientific Research Institute of Municipal Hygiene

Ul. Kirova, 6, Kiev, Ukr. SSR

Leningrad Institute of Radiation Hygiene Ministry of Health, RSFSR Ul. Mira, 6/8, Leningrad P-101

Leningrad Medical Institute for Sanitation and Hygiene Ministry of Health, RSFSR, Leningrad 67

Leningrad Scientific Research Institute of Industrial Hygiene and Occupational Diseases Ministry of Health, RSFSR 2-ya Sovetskaya ul., 4, Leningrad

Central Institute for the Advanced Training of Physicians Ministry of Health, USSR Ploshehad Vostania, No. 1/2, Moscow

Central Scientific Research Institute of Sanitation and Hygiene im. Erisman Academy of Medical Sciences, USSR

Pyatnikskaya ul., 1/2, Moscow, B-35 Institute of General and Communal Hygiene

Academy of Medical Sciences, USSR Pogodinskaya ul., 10, Moscow, G-117 Institute of Industrial Hygiene and Occupation

Institute of Industrial Hygiene and Occupational Diseases Academy of Medical Sciences, USSR Ul. Obukha, 14, Moscow

Moscow Medical Institute I im. B. Pirogovskaya 2/6, Moscow, G-48

Ufa Institute of Industrial Hygiene and Occupational Diseases Pervomayskaya ul., 20, Ufa, Bashkir ASSR, RSFSR

# COURSES FOR ENGINEERS AND CHEMISTS

THE DIVISION OF OCCUPATIONAL HEALTH, U. S. Public Health Service, announces a two-week, basic course for industrial hygiene engineers and chemists to be held at the Occupational Health Research and Training Facility, 1014 Broadway, Cincinnati 2, Ohio. The course will begin on January 30, 1961. Following the basic course there will be a one-week advanced course in noise measurement and control for engineers, and a similar course in lead analyses for chemists. These advanced courses will be held concurrently during the week of February 13–17, 1961. Applications should be addressed to the Facility at the above address, attention of the Chief, Training Operations Section. Sample schedules are available upon request.

Attendance is limited to 12 engineers and 12 chemists. In the event the courses are over-subscribed, applicants not accommodated will be given preference in June 1961 when the basic course will be repeated.

Applicants accepted will receive a copy of the training manual and other literature pertaining to the course. No tuition is charged for these courses. Travel and living expenses must be borne by the student or his employer.

# A Spectrophotometric Method for the Determination of Mercaptans in Air

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THIS method for the spectrophotometric determination of microgram quantities of mercaptans in air has been developed for air pollution and industrial hygiene studies. The mercaptans are collected in 5% mercuric acetate and reacted with N,N-dimethyl-p-phenylenediamine to form a red complex. The spectrophotometric sensitivity as defined by Sandell' at 500 m $\mu$  is 0.011  $\mu$ g of CH<sub>3</sub>SH/cm² or 0.00023 micromole of mercaptan/cm². The calibration curve follows Beer's law. H<sub>2</sub>S does not interfere with the determination.

In the determination of H<sub>2</sub>S by the methylene blue method, mercaptans were found to interfere by producing a pink color.<sup>2, 3</sup> Sliwinski and Doty<sup>4</sup> utilized this reaction to determine microquantities of methyl mercaptan in gamma-irradiated meat. Kusian, Pate, and Lipera<sup>5</sup> used the reaction to determine ethyl mercaptan. Investigations in this laboratory show that this reaction takes place not only with methanethiol and ethanethiol but also with n-butanethiol and n-hexanethiol. In addition, the reaction appears to take place with at least all of the low molecular weight alkanethiols, and is suitable for their determinations in air.

# Reagents

Lead methyl mercaptide. Prepare the crystalline solid by bubbling commercial CH<sub>8</sub>SH gas into a 10% lead acetate solution. Collect the yellow crystals by suction filtration, wash with distilled water and dry overnight in a vacuum oven at 45°C. Store crystals away from light and air. One mole of this mercaptide is equivalent to two moles of a mercaptan.

Mercuric acetate solution, 5%. Dissolve 50 gm of mercuric acetate, free of mercurous salts, in approximately 400 ml water and 25 ml glacial acetic acid. Dilute to one liter.

Lead methyl mercaptide, stock standard solution. Weight out 156.6 mg of the crystalline lead methyl mercaptide and make up to 100 ml with the 5% mercuric acetate. This solution contains the equivalent of 500  $\mu$ g of CH<sub>3</sub>SH per ml.

Lead methyl mercaptide, working standard solution. This solution contains the equivalent of  $10~\mu g$  CH<sub>3</sub>SH per ml and is prepared by the proper dilution of the stock standard solution with the 5% mercuric acetate solution.

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Amine solution. Dissolve 5.0 gm N,N-dimethyl-p-phenylenediamine hydrochloride in one liter of concentrated hydrochloric acid. When protected from light the solution is stable for at least six months.

Reissner solution. Dissolve 67.6 gm ferric chloride hexahydrate in distilled water, dilute to 500 ml and mix with 500 ml nitric acid solution containing 72 ml boiled concentrated nitric acid (specific gravity 1.42). This solution is likewise stable.

Color developing reagent. To three volumes of the amine solution add one volume of Reissner solution and mix. This solution should be freshly prepared for each set of determinations.

## Sampling and Analysis

Place 15 ml of the 5% mercuric acetate solution in a tall-form extra course fritted glass bubbler or a MSA midget impinger flask fitted with a coarse porosity frit, illustrated in Figure 1. Connect the bubbler between a pump and flowmeter and sample at a rate of 0.5 to 1.5 liters per minute. The maximum sampling rates for the midget impinger and tall-form bubbler described are approximately 0.6 and 1.5 liters per minute respectively. Where a power source is not available, the sampling may be done by liquid displacement from two polyethylene bottles of approximately 10 liters capacity.

Quantitatively transfer the sample from the collecting apparatus to a 25 ml volumetric flask and dilute to approximately 22 ml with distilled water that has been used to rinse the fritted bubbler and flask. Add 2.0 ml of freshly prepared color developing reagent, dilute to volume with distilled water and mix well. After 30 minutes read the color intensity at 500 m $\mu$  by means of a spectrophotometer.

Determine the mercaptan present by reference

to a calibration curve prepared by pipetting appropriate aliquots of the lead methyl mercaptide working standard into a series of 25 ml volumetric flasks, diluting to about 15 ml with 5% mercuric acetate and treating in the same manner as the sample. The blank is prepared in the same manner as the standards with the exception that no lead methyl mercaptide is added. The results may be reported as mercaptans calculated as CH<sub>9</sub>SH.

# Discussion and Results

It is not convenient to use methanethiol, ethanethiol, n-butanethiol or n-hexanethiol as primary standards in routine analytical procedures. Their odors and volatilities make the preparation of standards difficult and standard solutions are unstable. This instability of mercaptans has been pointed out in the literature.<sup>5</sup>

Other mercaptides have been tried as standards but lead methyl mercaptide has been the most satisfactory. This compound is easy to prepare and it is stable. In our experience it has not been necessary to carry out the recrystallization of the lead methyl mercaptide as was done by Sliwinski and Doty.4 Elemental analysis of the sulfur and lead content of a sample of the mercaptide prepared as directed above indicated that the product was of sufficient purity to be used as a standard. A comparison of the results of an elemental analysis of a recrystallized sample prepared by Sliwinski and Doty and one prepared in this laboratory without recrystal-lization is given in Table I. The sulfur content was determined by dissolving a sample of the mercaptide in a 0.1M solution of the disodium salt of ethylenediaminetetraacetic acid (EDTA) and 95% ethanol and then analyzing by the method of the Institute of Petroleum.<sup>10</sup> The lead content was determined gravimetrically as

Mercuric acetate used in this procedure must be free of mercurous ion in order to prevent the precipitation of Hg<sub>2</sub>Cl<sub>2</sub> during the color development. Even reagent grade mercuric acetate sometimes contains mercurous mercury. The suitability of the mercuric acetate may be ascertained by adding 3 ml of concentrated HCl to 15 ml of the 5% solution of mercuric acetate. If the solution becomes cloudy upon addition of the HCl the mercuric acetate is not suitable.

A mercuric nitrate solution prepared by dissolving 50 gm of Hg(NO<sub>3</sub>)<sub>2</sub>·H<sub>2</sub>O, free of mercurous mercury, in approximately 300 ml of 0.2N HNO<sub>3</sub> and diluting to one liter may be substituted for the 5% mercuric acetate solution throughout this procedure except in the prepara-

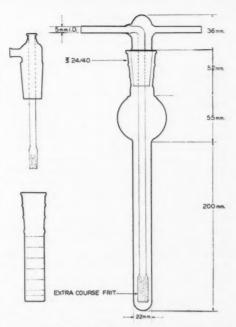


FIGURE 1. Unassembled midget impinger flask with fritted bubbler and assembled tall-form bubbler used for sampling mercaptans in air.

Table I
Comparison of Recrystallized and
Non-recrystallized Lead Methyl
Mercaptide

	%Pb	%S	%C	%H
Theoretical	68.75	21.27	7.97	2.01
Recrystallized sample of Sliwinski & Doty	69.0	20.9	8.00	2.06
Non-recrystallized sample	68.6	21.1	-	-

tion of the lead methyl mercaptide stock standard solution.  $Pb(SCH_3)_2$  is insoluble in the mercuric nitrate solution.

The minimum amount of a mercaptan that can be determined will depend upon its molecular weight and the length of the light path available for the colorimetric determination. With a 50 mm absorbance cell one can readily determine from 1 to 50 micrograms of CH<sub>8</sub>SH, 0.021 to 1.0 micromoles of mercaptan. This gives a working range of 0.5 to 25 ppm of CH<sub>8</sub>SH for a one liter air sample. Samples in this working range are readily determined with the Beckman Model B or a similar spectrophotometer; however, a colorimeter equipped with the proper

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TABLE II
Odor Thresholds of Some Alkanethiols

Alkanethiol	μg/liter	ppm
Methanethiol	1.1	0.56
Ethanethiol	0.013	0.05
n-Propanethiol	0.075	0.38
Butanethiol	1.4	0.38

filter may also be used. The sensitivity for a Klett photoelectric colorimeter with No. 54 Klett filter was found to be 0.0006 micromole of mercaptan/cm² or 0.029 µg methyl mercaptan/cm².

The intensity of the red complex formed with the alkanethiols is proportional to the mercaptan sulfur present and thus the sensitivity of the method is inversely proportional to the method is inversely proportional to the molecular weight of the alkanethiol. It follows that on a weight basis larger amounts of the higher molecular weight compounds must be sampled in order to be determined. The lower sensitivity to the higher molecular weight compounds has not proved to be a practical disadvantage because the odorous low molecular weight alkanethiols are of primary interest in industrial hygiene and air pollution studies.

Occasionally one can smell a mercaptan in air and yet after sampling five to ten liters of air is unable to detect any mercaptan by the procedure given here. This is due to the fact that the odor thresholds for various alkanethiols differ and that the sampling time must be adjusted accordingly. Odor thresholds taken from the literature<sup>5, 11</sup> are given for several alkanethiols in Table II. Assuming that the minimum determinable amount of methanethiol is one  $\mu g$ , one can see that only one liter of air need be sampled to detect methanethiol in concentrations as low as its odor threshold, but under the same conditions one must sample nearly 80 liters of air to determine ethanethiol in concentrations equal to its odor threshold.

In the range 410 m $\mu$  to 650 m $\mu$  the absorption spectra of the red complex formed by the reaction of N,N-dimethyl-p-phenylenediamine with lead methyl mercaptide, methanethiol, ethanethiol, n-butanethiol, and n-hexanethiol are almost identical (Figure 2). This emphasizes the fact that with the procedure described above one can only determine total mercaptans and cannot differentiate between the individual alkanethiols found in a mixture.

The accuracy and reproducibility of the method was tested by determinations on Eastman White Label alkanethiols. The alkanethiols were weighed out and dissolved in 95% ethanol. The alcoholic solutions were then diluted with the 5% mercuric acetate solution to the desired concentrations and the determinations made. A summary of the data is given in Table III.

To test the collection efficiency and repro-

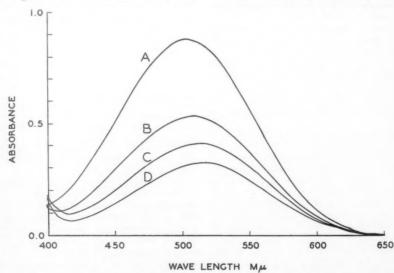


FIGURE 2. Absorption of complex formed by lead methyl mercaptide and some mercaptans with N,N-dimethyl-p-phenylenediamine. (A) Lead methyl mercaptide; (B) Ethanethiol; (C) N-Butanethiol; and (D) N-Hexanethiol.

ducibility with simulated field concentrations of mercaptan, a dilute stream of ethanethiol was generated by the diffusion bulb method.12 Eastman White Label ethanethiol, diluted in 95% ethanol, was used as a source of mercaptan in the diffusion apparatus to give dilute streams of mercaptan in air in the desired concentration range. Samples were collected by two bubblers connected in series. Results are given in Table IV. For data shown, the flowrate of the carrier gas (air) was 0.5 liter per minute and the sampling time was 10 minutes. The mercaptan concentrations were  $4.3 \pm 0.4$  ppm and  $0.34 \pm 0.02$ ppm calculated as CH3SH. A variation of 5 to 10% from the mean concentrations due to the variability of sampling, analysis and diffusion rate was found. A collection efficiency of 100% for ethanethiol is indicated by the fact that no mercaptan was found in the second bubbler. To test the collection efficiency of methanethiol, commercial tanked CH3SH gas was diluted with air to approximate concentrations of 100 and 200 ppm and sampled through a series of two bubblers. No mercaptan was detected in the second bubbler and it was concluded that the collection efficiency for methanethiol was also

The effect of hydrogen sulfide, sulfur dioxide, and nitrogen dioxide on the determination of mercaptans was investigated. Up to 100 μg of H<sub>2</sub>S caused no detectable effect when equal volumes of dilute H<sub>2</sub>S and CH<sub>3</sub>SH gas were sampled and analyzed by this procedure. No color change was detected when 250 μg of SO<sub>2</sub> was sampled from a stream at a concentration of 300 ppm.

To test the effect of  $NO_2$  on sampling and analysis of mercaptan,  $NO_2$  at various concentrations was sampled with the collecting reagent followed by addition of 50  $\mu g$  of  $CH_9SH$  as  $Pb(SCH_3)_2$ . The results were the same whether the mercaptan was added before or after sampling the  $NO_2$  stream. The results are summarized in Table V. Analysis for  $NO_2$  showed that this gas is very inefficiently collected by the mercuric acetate solution. No appreciable effect was observed when up to  $700~\mu g$  of  $NO_2$  was sampled at one liter per minute from an atmosphere containing 6 ppm of  $NO_2$ . Higher concentrations of  $NO_2$  caused a positive interference.  $NO_2$  sampled in the absence of mercaptans did not give a positive value for mercaptans.

This method has been used successfully in the field to determine mercaptans quantitatively wherever they could be detected by odor. Where mercaptans could not be detected by odor they were not detected by this method.

Table III
Accuracy and Reproducibility of Results of
Analyses of Some Alkanethiols

Alkanethiol	Number of deter- mina- tions	Amount present,		Error, per cent	Coeffi- cient of varia- tion, %
Ethanethiol	6	199	199	0	0.8
Butanethiol	6	208	202	-3	0.6
Hexanethiol	6	210	197	-6	2.0

Table IV

Collection Efficiency and Reproducibility of
Determinations of Mercaptans in Air at
Field Concentrations

Concentra-	Number of	First B	Second Bubbler	
captans in air as CH <sub>2</sub> SH, ppm	determina- tions	Mean amount mercaptan found, μg	Coefficient of varia- tion, %	Mercaptan found, μg
4.3	8	101	7.1	0
0.34	6	8.1	2.6	0.0

Table V
Interference of Nitrogen Dioxide on the Determination of Mercaptan

NO <sub>2</sub> ppm	Range of quantity of NO <sub>2</sub> sampled, µg	Coefficient of variation for mercaptan determinations, %	Error in mer- captan determi- nations, %	Interpretation
1	7-131	2.4	-4	No effect detected
6	58-710	6.5	+6	66 66 66
14	133-663	4.5	+20	Results high
33	62-310	6.6	+31	66 66
38	72-720	9.8	+27	66 66

# Conclusions

The method described is quick and accurate and determinations may be made using apparatus common in air pollution and industrial hygiene work. The method determines total mercaptans and does not differentiate between individual mercaptans although it is most sensitive to the low molecular weight alkanethiols. H<sub>2</sub>S, SO<sub>2</sub>, and NO<sub>2</sub> do not interfere at the concentrations usually encountered in air pollution and industrial hygiene studies.

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# INDUSTRIAL HYGIENE FELLOWSHIPS

THE ATOMIC ENERGY COMMISSION is now accepting applications for AEC Fellowships in Industrial Hygiene for the academic year 1961–1962. Applicants must have a bachelor's degree with major in physics, chemistry or engineering. Mathematics through calculus is required and some biology is desirable. Fellows must be United States citizens, under 35 years of age, and acceptable for graduate work at the university to which they are assigned. Tenure is for the academic year.

Fellows will receive training in industrial hygiene at Harvard University, the University of Cincinnati, the University of Michgan, or the University of Pittsburgh. Assignments to institution of choice will be made insofar as possible, but cannot be guaranteed. All fellows are required to take courses in public health and biostatistics. Course work will vary with the different institutions, but latitude for elective courses is allowed. Information regarding course requirements for degrees can be obtained from the several universities.

The basic stipend is \$2500. An additional \$350 is allowed for a spouse and \$350 for each dependent child. Normal tuition and fees as well as limited travel allowance are paid by the fellowship. Fellows with graduate work or industrial experience may be eligible for an additional allowance. Fellows will be assisted financially to attend the annual meeting of the American Industrial Hygiene Association. Acceptance of these fellowships involves no commitment with regards to future employment.

Completed applications, supporting letters of reference, and transcripts must reach Oak Ridge Institute of Nuclear Studies not later than March 1, 1961. Forms and detailed information may be obtained from the Industrial Hygiene Fellowship Office, Oak Ridge Institute of Nuclear Studies, Oak Ridge, Tennessee.

# The Determination of n-Butylamine in Air

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# Introduction

The total production of aliphatic amines in 1958 was about 260 million pounds. Of this total, 667,000 pounds were n-butylamine. This compound is finding increasing use in the manufacture of such diverse products as dye stuffs, rubber chemicals, flotation agents, corrosion inhibitors, petroleum specialities, and insecticides and is an ingredient of some photographic processing solutions. Despite the relatively widespread use of this chemical, it has been subject to little investigation from the standpoint of industrial hygiene.

n-Butylamine is a clear liquid which tends to yellow on standing and has a characteristic ammoniacal odor. It is flammable and has an open cup flash point of 45°F.<sup>3</sup>

# **Physiological Effects**

Insofar as it affects human health, the principle hazard of concentrated n-butylamine is its ability to produce severe burns of skin or eye. It is interesting to note that in 1923, Hanz-lik reported that the normal butylamines are non-irritating to human skin.

Hanzlik also reported a central nervous stimulation, convulsions, then depression and narcotic death with pulmonary edema. Smyth states that the rat, when exposed to butylamine vapor, survives four hours at 2,000 ppm but dies from 4,000 ppm. Our own laboratory has found that three out of three rats died in a 50-minute exposure to 3,150 ppm. A concentration of 2,500 ppm killed three out of three rats; one in 12 hours, one in two days, and one in three days.

Smyth<sup>6</sup> feels that the most important effect of n-butylamine inhalation is respiratory tract irritation, with lung edema the maximum injury. The current MAC of 5 ppm is probably based on irritative action. Our own observations made in the field while sampling this material showed that an air concentration of 0.12 ppm has a very faint odor, while levels from 3 to 10 ppm were characterized as having a strong odor.

Presented at the Twenty-first Annual Meeting of the American Industrial Hygiene Association, Rochester, New York, April 28, 1960.

Exposure to n-butylamine vapors may result in erythema, particularly about the face. One of the authors (RFS) showed the following reactions after short exposures to only a few ppm of butylamine. Face and neck became florid within three hours after exposure; desquamation occurred on the facial skin three days following exposure; a burning, itching sensation accompanied these symptoms. Such symptoms, however, have not been noted in other exposed personnel.

# Determining n-Butylamine in Air

The vapors of n-butylamine could be sampled by drawing the contaminated air through bubblers containing measured volumes of dilute standard sulfuric acid. The solution would then be titrated with standard caustic. This method is far from satisfactory. The concentrations of n-butylamine encountered in plant air are often below 5 ppm and an extremely long sampling period is required to detect such concentrations by a titrimetric method. Also, such a method is subject to interference by all airborne basic materials, such as ammonia and the other alkyl amines. More often than not in our plant, these materials were used in the same areas with n-butylamine.

Davies, Wolfe, and Perry developed an extremely sensitive colorimetric method for detecting the quantities of amines present in biological fluids.7 The method depends on the reaction of the amine with ninhydrin (1,2,3-triketohydrindene), a reagent commonly used for amino acids. Figure 1 shows the reaction, omitting the intermediate steps.8 This method has proved to be well adapted to the requirements of air analysis. It is extremely sensitive and is sufficiently specific to distinguish n-butylamine in the presence of many substances which would interfere with the method based on absorption in sulfuric acid. A one cubic foot air sample, drawn through 10 ml of absorbing solution, will detect 0.1 ppm of butylamine vapor.

Reagents Required for Analysis

1. Ninhydrin reagent. Prepare a 0.2% solution of ninhydrin (E.K. No. 2495) in isopro-

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Figure 1. Reaction between n-butylamine and ninhydrin.

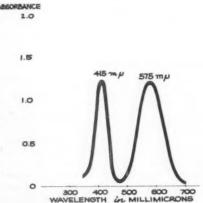


FIGURE 2. Visible light absorption spectrum of n-butylamine-ninhydrin reaction product.

panol (0.158 gm diluted to 100 ml with isopropanol). This reagent should probably be used within two weeks of preparation.

2. Acid-isopropanol mixture. A solution made by diluting one volume of concentrated hydrochloric acid to 100 volumes with isopropanol. Two liters of this reagent is a convenient quantity to make up.

3. n-Butylamine stock solutions. (N.B. All dilutions are made with acid-isopropanol mixture.)

Concentrated Stock solution (500 gammas/ml)
Dilute 0.338 ml of n-butylamine to 500 ml.

Solution No. 1 (100 gammas/ml) Dilute 20 ml of the concentrated stock solution to 100 ml.

Solution No. 2 (50 gammas/ml)

Dilute 5 ml of Solution No. 1 to 10 ml.

Solution No. 3 (25 gammas/ml)

Dilute 2.5 ml of Solution No. 1 to 10 ml.

Solution No. 4 (10 gammas/ml)

Dilute 1.0 ml of Solution No. 1 to 10 ml. Solution No. 5 (5 gammas/ml)

Dilute 0.5 ml of Solution No. 1 to 10 ml. Solution No. 6 (2 gammas/ml)

Dilute 0.2 ml of Solution No. 1 to 10 ml.

# Sampling

Measured volumes of the vapor-laden air are drawn through acid-isopropanol solution contained in either gas washing bottles or midget impinger flasks with the fritted glass bubbler. Two bottles may be used in series, although our tests have shown that absorption is essentially complete in the first bottle. The samples may be kept in glass stoppered bottles and have shown no deterioration over fairly prolonged periods (at least three weeks).

# Analytical Procedure

- 1. Measure volume of sampling solution.
- 2. From each sample a proper aliquot (no more than 3 ml) is transferred to a numbered glass-stoppered test tube.

To another tube is added 3 ml of the acidisopropanol mixture. This tube serves as the reagent blank.

Next, 1 ml each of stock solutions No. 1 through No. 6 are pipetted into separate glass-stoppered test tubes. These standards are treated exactly as the samples.

3. 5 ml of pyridine, 2 ml of ninhydrin reagent and enough acid-isopropanol to make a final volume of 10 ml are then added to each sample, standard, and blank tube.

4. The tubes are stoppered and after thorough mixing inserted in a water bath at 85°C (185°F) for 7 minutes. Avoid contact of the solution with the skin.

5. After 7 minutes the tubes are removed from

the water bath and immersed in cold running water for about 10 minutes.

6. The absorbance is then read at 575 m $\mu$  using a one centimeter cell in a suitable colorimeter with a tungsten light source. Figure 2 shows a typical absorption spectrum resulting from 200 micrograms of n-butylamine in the aliquot used for color development.

7. The butylamine content of the sampling solution is then determined. The absorbance obtained by developing a color in a suitable aliquot of these solutions is compared with a curve prepared by plotting the absorbances obtained from the known quantities of n-butylamine.

8. The ppm in the original air sample is calculated as follows:

$$\mathrm{ppm} = \frac{\mathrm{P} \times \mathrm{F} \times 25 \times 10^6}{73.14 \times \mathrm{V} \times \mathrm{M} \times 28320}$$

Where: ppm = cubic centimeters of n-butylamine vapor per cubic meter of air.

P = total milligrams of n-butylamine in the 10 milliliters of colored solution.

F = The following ratio: total milliliters of sampling solution/ milliliters of sampling solution used as aliquot.

25 = cubic centimeters of vapor in one millimole of any substance at room temperature (approximate).

73.14 = millimolecular, weight of butylamine, milligrams.

V = sampling rate in cubic feet per minute.

M = length (minutes) of the air sampling period.

28320 = factor used to convert cubic feet to cubic centimeters.

## Comments

As shown in Table I, the ninhydrin reagent is relatively stable at least for a one month period. As a precautionary measure, we generally make this solution up fresh for each analysis.

Table II shows the absorption efficiency of the acid-isopropanol sampling solution and indicates that for all practical purposes, recovery is complete in one gas-washing bottle.

Table III indicates that the n-butylaminehydrochloric acid salt is stable at least over a three-week period. This permits postponement of analysis until convenient.

Table I Stability of Ninhydrin Reagent

Concentration of n-butylamine in solution (ppm)	Optical density on various days (ninhydrin reagent made on day 1)			
	(day)			
	1	6	26	
5	0.041	0.041	0.041	
10	0.073	0.073	0.086	
25	0.200	0.210	0.215	
50	0.336	0.365	0.335	
100	0.688	0.635	0.710	

Table II

Absorption Efficiency of Acid-Isopropanol
Sampling Solution

Butylamine concentration (ppm)	Recovery in 1st bottle* (%)
5	100.0
25	100.0
100	100.0
2450	99.9
5450	99.8
11450	99.6

\* Two gas washing bottles were used in series. Each contained 30 ml of acid-isopropanol solution.

TABLE III

Optical Density Obtained by Analyzing Various Concentrations of n-Butylamine in Acid-Isopropanol Solution

Concentra- tion of n-butylamine	Days after preparation of solution					
in solution (ppm)	0	5	12	25		
5	0.041	0.041	0.041	0.041		
10	0.073	0.073	0.070	0.085		
25	0.200	0.210	0.210	0.214		
50	0.336	0.365	0.360	0.336		
100	0.688	0.635	0.665	0.710		

Table IV Stability of n-Butylamine-ninhydrin Complex

Concentra- tion of n-butylamine	Optical density at various times (out of ice bath at 0:00)				
in solution (ppm)	Time	O.D.	Time	0.D.	
5	0:02	0.041	1:17	0.029	
10	0:03	0.073	1:18	0.060	
25	0:04	0.200	1:19	0.181	
50	0:05	0.336	1:20	0.305	
100	0:06	0.688	1:21	0.600	

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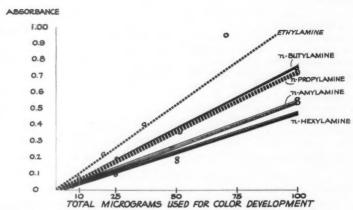


FIGURE 3. Absorbance (optical density) values of reaction products of ninhydrin with several alkylamines are plotted against known amounts of the alkylamines.

Table IV indicates that the n-butylamineninhydrin reaction product is not stable but fades fairly rapidly within a few hours.

Figure 3 plots absorbance (or optical density) versus micrograms of butylamine and some other alkyl amines which may be determined by this method. Although not shown on the curve, preliminary work indicates that, with slight modification, the method will be suitable for diethylene triamine.

# Acknowledgments

All unpublished toxicity data contained herein were obtained from R. L. Roudabush, Ph.D. and C. J. Terhaar, Ph.D., of Eastman Kodak Company's Laboratory of Industrial Medicine.

The visible light absorption spectrum for the butylamine-ninhydrin complex was prepared by

Stuart Foster of this company's Industrial Laboratory.

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# AIHA ABSTRACTS

FOM MANY SIDES WE HAVE RECEIVED indications and suggestions that many, even most, of our readers would like to have a brief summary or abstract of each article appear at the beginning of the article. The Board of Directors has looked favorably upon this idea so we are going to try it.

Authors will note that henceforth they should submit such an abstract with their manuscript. This abstract should be short, preferably not more than 100 words, and should indicate the scope of the article and its major conclusions or findings.

In the coming issues there will be a transition period when not all articles will be accompanied by the abstract. We trust that this period will be brief but as we "have a go at it" let us know what you think of it.

# **Determination of Mercury in Blood**

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# Introduction

IT IS well known that mercury and its compounds are widely encountered as occupational poisons. It is less well known that the laboratory methods for confirming the fact of occupational mercury poisoning or absorption are not adequate despite a relatively large amount of work in this field, particularly in determining urinary excretion of mercury. Reports, however, on mercury levels in blood have been limited in

It is the purpose of this study to develop a method for the determination of mercury in blood which is simple enough to encourage wide application. This is the initial report of our investigations. With this new method, it is our hope that it will be possible to make further studies on the correlation between mercury blood levels and the existence of clinical evidence of mercury poisoning and perhaps assist in the diagnosis of mercury intoxication before clinical signs develop. Our results will be detailed in future reports.

The methods for the determination of mercury in biologic materials usually require three types of manipulation: first destruction of organic matter generally by wet ashing; secondly the isolation of the mercury; and third the estimation of the isolated mercury. Representative of the first group of manipulative steps are the methods of Cholak and Hubbard, Barrett, Vesterberg and Sjöholm, Miller and Swanberg, and Monkman

Maffett, and Doherty.

Cholak and Hubbard1 used a method in which the organic matter was destroyed by boiling with a mixture of sulfuric and nitric acids followed by a second boiling with potassium permanganate. Barrett<sup>3</sup>, employing a combination of the Cholak and Hubbard method and that of Laug and Nelson, digested the biologic material with sulfuric and nitric acids, and with permanganate, using a reflux condenser to minimize losses of mercury during the heating step. Such losses were stressed by Simonsen.8

These studies were supported in part by grants from Lakeside Laboratories and National Institutes of Health, Public Health Service, Department of Health, Education and Wel-

Vesterberg and Sjöholm' wet ashed the sample with an acid solution of chlorine after separation of the mercury as the sulfide with cadmium sulfide as the collector, a second wet combustion with chlorate, hydrochloric acid, and chlorine was performed.

Polley and Miller used sulfuric acid and hydrogen peroxide, destroying excess peroxide with permanganate and the permanganate with hydroxylamine hydrochloride. Gettler and Lehman<sup>10</sup> used potassium permanganate with sulfuric and nitric acids, and Johansson and Urnell11 also used sulfuric acid and permanganate but destroyed the excess permanganate and manganese dioxide with peroxide and oxalic acid instead of the customary hydroxylamine hydrochloride.

Elkins12 suggests the use of hydrochloric acid and potassium chlorate, Other variations are those of Milton and Hoskins13 and Maren.14

Monkman, Maffet, and Doherty' employed cold digestion with solid potassium permanganate and concentrated sulfuric acid and permitted the mixture to stand overnight. Miller and Swanberg<sup>5</sup> also used cold digestion with hydrogen peroxide and a catalyst of ferric chloride and potassium chromium sulfate.

Campbell and Head<sup>15</sup> have critically reviewed a number of these types of wet digestion and recommend the use of potassium permanganate and sulfuric acid for the determination of mercury in urine.

Most methods utilize dithizone to isolate the mercury from the digestion mixture after adjustment to a pH of about one to avoid interference of other metals in the extraction. Hubbard16 and Cholak and Hubbard,1 however, used di-β-naphthylthiocarbazone for this purpose. Campbell and Head<sup>15</sup> following the technique of Vašák and Šedivec17 employed ethylenediaminetetraacetic acid, disodium salt to chelate interfering substances before the dithizone extraction.

Ballard and Thornton18 separated the mercury by filtration through a bed of asbestos fiber impregnated with cadmium sulfide, modifying the method of Clarke and Hermance19 who used filter paper impregnated with cadmium sulfide. Monk-

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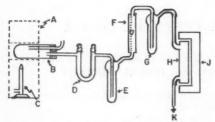


FIGURE 1. Diagrammatic sketch of the apparatus for the microdetermination of mercury: A—Wire enclosure of heating chamber, B—Ignition tube, C—Bunsen burner, D—U-tube containing absorbent cotton, E—Cold trap, F—Rotameter, G—Cold trap, H—Optical cell, J—Mercury vapor meter, K—To water aspirator pump.



FIGURE 2. Kruger mercury vapor meter modified by replacement of the grille with a special optical cell.

man, et al. modified this manipulative step additionally by impregnating glass filter pads.

Kozelka<sup>20</sup> separated the mercury after a digestion with sulfuric acid, copper sulfate and ammonium sulfate by distillation with chlorine. Barrett<sup>2</sup> as well as Laug and Nelson<sup>7</sup> used a second extraction with potassium bromide to free the mercury from interferences and Winkler<sup>21</sup> employed sodium thiosulfate. Storlazzi and Elkins<sup>22</sup> using a procedure modified after the method of Stock and Lux<sup>23</sup> separated the mercury by electrolysis.

In the third group of manipulative steps, namely, those for estimating the isolated mercury, representative variations are the colorimetric or spectrophotometric determination usually of a solution of mercury dithizonate at 490 m $\mu$  as done by Barrett, by Campbell, and Johansson and Urnell, colorimetric determination of the di- $\beta$ -naphthylthicarbazone complex, colorimetric estimation of cuprous mercuric iodide as done by Gettler and co-

workers;<sup>24, 25</sup> titrimetric estimation as in the methods of Locket<sup>20</sup> and of Nobel and Nobel;<sup>27</sup> microscopic estimation as detailed in the method of Storlazzi and Elkins;<sup>22</sup> and estimation by ultraviolet photometry based on the method of Woodson<sup>20</sup> by Ballard, et al.,<sup>26, 29, 20</sup> Monkman, et al.,<sup>26</sup> and Lindström.<sup>21</sup>

The method presented here, combining cold incomplete digestion, extraction with dithizone, decomposition of the mercury dithizone complex by heating to produce mercury vapor, and subsequent estimation of the mercury by ultraviolet photometry is relatively rapid and simple compared with the methods presently available. It is suitable for amounts of mercury in blood of the order of millimicrogram concentrations, that is, 10-° gram of mercury in one milliliter of blood.

# **Equipment and Reagents**

# Apparatus

The apparatus, Figure 1, comprises a heating chamber, connected to a U-tube containing absorbent cotton and followed by a cold trap, a rotameter, an additional trap, a modified Kruger mercury vapor meter Model 23 (Figure 2) and a water aspirator pump. The mercury vapor meter was modified by altering the grille so that it could be removed easily and be replaced by an optical cell. This cell is a cylinder 23 cm in length and 4 cm in diameter. It has removable silica end windows, and is fitted with inlet and outlet ports for the air stream drawn by the aspirator pump. One of our collaborators (Gilbert) proposed the use of such a cell in 1957. A calibrated Fischer and Porter rotameter equipped with both glass and steel floats was used to measure the air flow.

The heating chamber consists of a Bunsen burner mounted in a Nichrome wire cage  $3\frac{3}{4}$  x  $13\frac{3}{4}$  x 12 inches. Two cylindrical openings having a diameter of one inch capable of snugly accepting a 25 x 200 mm Pyrex ignition tube are cut in opposite faces of the unit about  $6\frac{1}{2}$  inches from the bottom and about 2 inches above the top of the burner. A 25-mm diameter wire cylinder fits between the two openings, and the ignition tube passes through this cylinder.

# Reagents

Potassium Permanganate Solution, 6%.—Dissolve 60 grams of reagent grade potassium permanganate in distilled water with the aid of heat, and dilute to one liter with distilled water.

Sulfuric Acid.—From concentrated reagent grade sulfuric acid prepare a 0.25N solution in the customary manner.

Dithizone Extraction Solution.—Weigh out accurately 6 mg of purified diphenylthiocarbazone

and dissolve in reagent grade chloroform. Transfer to a liter volumetric flask. Make to volume with chloroform.

Hydroxylamine Hydrochloride Solution, 20%. -Dissolve 60 grams of reagent grade NH<sub>2</sub>OH. HCl in 650 ml of distilled water and purify as follows: Add a few drops of m-cresol purple indicator solution. Add ammonium hydroxide solution until the mixture turns yellow. Add one ml of 5% sodium diethyldithiocarbamate solution and extract with chloroform until the metal complexes and all the excess diethyldithiocarbamate have been removed. Test for complete extraction of the reagent by the addition of a small amount of dilute copper solution to a small portion of the purified hydroxylamine hydrochloride reagent. If the extraction is complete no yellow color is produced. Add pure hydrochloric acid until the m-cresol purple indicator turns pink. Then transfer the purified reagent to a one-liter volumetric flask and dilute to volume with distilled water.

#### Standard Solutions

Standard Stock Mercury Solution.—Weigh out exactly 0.1354 gram of mercuric chloride and dissolve in 0.25N sulfuric acid. Transfer to a 100-ml volumetric and dilute to the mark with 0.25N sulfuric acid. This solution contains one mg of mercury per milliliter.

Standard Working Mercury Solution.—Transfer, with the aid of a pipette, one ml of the stock standard to a 100-ml volumetric flask. Dilute to the mark with distilled water. This solution contains 10 micrograms of mercury per milliliter. Transfer one ml of the  $10~\mu \rm g/ml$  solution to a 100-ml volumetric flask and dilute to volume with distilled water. This working standard solution contains 0.1 micrograms per milliliter. These solutions must be prepared fresh as required.

#### Preparation of Standard Curve

Place 250 ml of 0.25N sulfuric acid in a separatory funnel with a dry stem. Add one ml of the standard mercury solution containing 10 micrograms of mercury per milliliter and mix. Extract with 5 ml of dithizone extraction solution shaking for one full minute and allowing the phases to separate completely. Swirl the separatory funnel to make sure all of the chloroform solution is brought to the bottom of the funnel. Release the pressure of the chloroform through the mouth of the funnel and not through its stopcock. Transfer the clear extract to a 100-ml, glass-stoppered volumetric flask, being careful not to draw any of the aqueous layer into the

flask. Repeat the extraction twice with 5-ml portions of the dithizone extraction solution and then once more with 5 ml of chloroform. Add these extracts to the volumetric flask and then complete to volume with chloroform. Mix thoroughly. This solution contains 0.1 microgram of mercury per milliliter. Transfer with the aid of pipettes 0.2 ml of dithizone extraction solution to each of two 25 x 200 mm Pyrex combustion tubes to serve as the blank. Transfer 0.1, 0.2, 0.3, and 0.4-ml portions of the chloroform solution containing 0.1 microgram of mercury (as mercury dithizonate) per ml in duplicate to separate Pyrex combustion tubes. The tubes will then contain respectively 0, 0.01, 0.02, 0.03 and 0.04 microgram of mercury in duplicate. Place the tubes into a beaker of hot water and evaporate off the chloroform completely at 70-80°C. Continue as detailed in the procedure. Plot the maximum arbitrary scale reading in mg/m<sup>8</sup> against the micrograms of mercury of the standard to obtain the standard curve. An alternative method is to plot one-half the product of the time interval from the start of movement of the meter pointer to its return to zero by the maximum arbitrary scale reading in mg/m3 against micrograms of mercury in the standard. The average curve obtained with results for five series of tests are shown in Figure 3 and Table 1.

#### Procedure

Transfer one ml of blood with the aid of a pipette to a 125-ml, glass-stoppered Erlenmeyer flask. Dilute with 20 ml of water. Add 10 ml of 6% potassium permanganate solution and add cautiously 2 ml of concentrated sulfuric acid and swirl. Warm on a hot plate set at low heat (do not boil), remove from hot plate, swirl, and allow to stand for 15 minutes. Repeat the warming on the hot plate, removing, swirling, and cooling two more times. Allow to stand until the super-

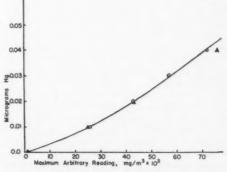


FIGURE 3. Standard curve.

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TABLE I Standard Curves

Mercury	No. of Detns.	Maximum Arbitrary Scale Reading, mg/m <sup>3</sup> × 10 <sup>2</sup>			
μg		Avg.	Max.	Min	
0	8	1	3	0	
0.01	9	26	34	23	
0.02	11	43	54	32	
0.03	3	57	68	48	
0.04	8	72	84	68	

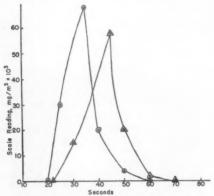


FIGURE 4. Comparative areas of mercury determinations.

natant solution is clear or for a total elapsed time of one hour. Dissolve the precipitate with 0.5 ml of 20% hydroxylamine hydrochloride solution. Swirl to assist in dissolving the precipitate. Stopper the flask, shake, cautiously release the pressure, and allow to stand for 10 minutes. Add 20% hydroxylamine hydrochloride solution drop by drop if more is required to dissolve the precipitate.

Transfer the solution to a dry 125-ml separatory funnel with a dry stem. Wash the Erlenmeyer flask with three 10-ml portions of distilled water, add the washings to the separatory funnel, and mix. Add 5 ml of the dithizone extraction solution. Stopper the separatory funnel and shake vigorously for one minute. Release the pressure through the mouth. Swirl to bring down any chloroform droplets and allow to stand until the phases separate completely. Draw off the chloroform layer into a glass-stoppered, 25-ml graduated cylinder, being careful not to draw off any of the aqueous layer. Repeat the extraction with 5 ml more of dithizone extraction solution and then wash with 3 ml of pure chloroform. Add the chloroform wash to the graduated cylin-

der. Add sufficient chloroform directly to the cylinder to make the total volume 15 ml.

Transfer with the aid of a pipette and a safety pipetter, one ml of the chloroform solution of mercury dithizonate to a Pyrex ignition tube 25 x 200 mm and place in a hot water bath. Evaporate off the chloroform completely at 70–80°C.

While the chloroform is being evaporated check the apparatus, turn on the Kruger mercury vapor meter, turn on the aspirator pump so that the air flow is about 3300 ml per minute, and allow the instrument to come to equilibrium. Check the span of the instrument. After all the chloroform has evaporated, dry the outside of the tube and insert the ignition tube into the heating unit placing it so that the flame will heat the bottom end of the tube. Light a match. insert it into the lighting opening, turn on the gas, allow it to ignite, remove the match, and start a stop watch. Record the starting time, in seconds, of the movement of the meter needle. the maximum deflection of the meter needle, the meter readings at 10 second intervals, if possible, and the time of return of the meter needle to zero. Obtain the concentration from the standard curve.

Multiply this reading by 15 to obtain the concentration in micrograms of mercury in one ml of blood and subsequently by 100 to express the result in micrograms per 100 ml of blood.

#### Results

In order to evaluate the method, known concentrations of mercury were added to blood samples. Each sample was then analyzed by the method detailed in the procedure. The results obtained by the addition of 0.1, 0.2 and 0.3 microgram of mercury to one ml blood samples for two series of experiments with blood are shown in Table 2. The results obtained with known concentrations of mercury for preparation of the standard curve have been given in Table 1 in the section on the preparation of the standard curve.

#### Discussion

Most of the methods presently used for the determination of mercury in blood require at least 10 ml of blood and preference is expressed for 20 ml. The requirement of only one ml in the method presented here is a marked advantage. Since only one ml of a total volume of 15 ml of chloroform solution is used for the photometric step, even smaller volumes of blood can be analyzed by a simple modification of the method in which smaller volumes of reagents and a smaller final volume are employed.

The isolation of mercury by means of extraction with dithizone serves not only to separate it from other metals but also serves to free it from most other materials so that in the heating step no interferences such as those elaborated by Lindström<sup>31</sup> are encountered. Furthermore, since a type of destructive distillation is used, no products of combustion such as those produced in the flame combustion variation of Lindström are introduced into the photometer cell.

A cold digestion similar to that of Monkman, et al. was followed with two variations. First, a solution of potassium permanganate was used instead of solid permanganate. This circumvented the need to raise the temperature to dissolve the crystals or pellets and, in addition, the reaction time was reduced because the permanganate was able to react immediately. Secondly, no attempt was made to destroy the organic matter completely. It was shown by Jacobs 32, 23 years ago that it was not necessary to destroy all the organic matter to determine the amount of arsenic in milk. Nobel and Nobel27 also do not destroy the organic matter completely in their method for the determination of mercury in urine. McBryde and Williams34 eliminated the digestion for urine samples completely, filtering them directly through cadmium sulfide impregnated pads. It is unlikely that such a variation would work with blood. Weiner and Müller35 have stressed the need for adequate digestion in order to avoid the interference of sulfhydryl groups.

The avoidance of high temperature in the digestion (not over 50-60°C) and the elimination of the use of nitric acid are advantageous; indeed, the elimination of the need of relatively large amounts of sulfuric acid, nitric acid, and potassium permanganate is a great advantage but, in addition, the elimination of nitric acid avoids subsequent oxidation of dithizone.

In this method we have tentatively adopted the convention of taking the maximum deflection of the meter pointer as representative of the concentration of mercury. This follows the variation of Yamaguchi<sup>35</sup> and is somewhat similar to the convention adopted by Lindström<sup>31</sup> in taking a reading at exactly 45 seconds. Reading the maximum meter deflection to obtain the concentration of mercury serves very well for the standard curve and for concentrations less than 0.02 microgram. There is greater variation for concentrations above 0.02 microgram in both the standard and blood determinations.

The recovery of mercury from the blood as shown in Table 2 varies from excellent to fair when the convention of reading the maximum meter deflection is observed. Actually the re-

TABLE II Recovery of Mercury from Blood

Mercury added µg	Max. Reading Mg/m <sup>3</sup> × 10 <sup>3</sup>	Reading minus blank	Concn. from Std. Curve
0	26		
0.0066	42	18	0.007
0.013	52	26	0.010
0.02	55	29	0.013
0	25		
0.0066	39	14	0.006
0.013	41	16	0.007
0.02	63	38	0.0018

covery of mercury is better than appears from the results using the maximum deflection variation. This may be shown by a comparison of the areas representing the total amount of mercury distilled. For example, in a specimen of blood to which 0.3 microgram of mercury was added to one ml, there is 0.02 microgram of added mercury in a one-ml aliquot of the 15 ml final chloroform solution. Two tests of this specimen gave maximum readings of 68 and 58 respectively for an average maximum of 63. Thus each value differed by 5 out of 63. When a plot of the time against meter deflections was prepared, Figure 4, and the areas were compared, the areas were in the ratio of 924 to 913, that is a difference of 6 in 918.

If the area is approximated by use of the equation

Area = 
$$\frac{1}{2}bh$$

where h is equal to the maximum deflection of the meter needle (i.e. the height of the rough triangle) and b is the time which elapses from the start of meter needle movement to its return to zero (i.e. the base of the rough triangle) the ratio of the results is 2380 to 2320 or roughly 3 out of 250. It is clear that the efficiency of recovery of mercury is greater than shown by some of the results using the maximum deflection variation.

We are planning to use a recorder to record the meter deflections. In this case the area under the curve on the chart can be used to calculate the concentration as was done by Monkman and co-workers using a General Electric Instantaneous Vapor Detector. We are also attempting to adapt the G. E. meter for our method. McBryde<sup>34</sup> has used a counter.

Most methods for the determination of mercury in blood currently used are time-consuming, requiring a number of days. Our method is relatively rapid for it can be completed in three hours.

Our work indicates that separation by extraction is superior to separation by precipitation as mercuric sulfide by passage of a test solution through cadmium sulfide impregnated papers, glass fiber, or asbestos.

We have tried the direct destructive distillation of 0.1 ml of blood to obtain the mercury vapor, using samples of blood both with and without added mercury. This variation appears to have possibilities. It will require a much more elaborate train for removal of aerosols than we now use or a system analogous to that of Ballard, et al., who used a spectrophotometer to measure the organic substances and applied a correction to their mercury reading. Such a variation would have the advantage of eliminating the acid digestion and the extraction steps.

We have made some preliminary tests indicating that we may be able to determine the amount of mercury directly in samples of the breath of individuals. Such samples were taken by aid of balloons and were passed through the apparatus. Reading in the millimicrogram range were obtained.

We expect to pursue this investigation vigorously applying it to breath, urine, body tissues, food, and other materials as well as to blood.

#### Summary

A method for the ultramicro-determination of mercury in blood has been presented. It depends upon a cold digestion of the blood with potassium permanganate and sulfuric acid, separation of the mercury by a dithizone extraction, liberation of mercury vapor by the destructive distillation of the mercury dithizonate, and estimation of the mercury by use of an ultraviolet light mercury vapor meter. Only one milliliter of blood is required for a determination. The method is suitable for the milli- and centimicrogram range, that is for 10<sup>-8</sup> to 10<sup>-9</sup> gram of mercury.

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## A Criterion for Evaluation of Noise Exposures

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#### **Earlier Criteria**

NOISE problems in the industrial environment are increasing in importance and scope. An effective hearing conservation program is one which includes workers with significant noise exposure but excludes those whose exposure is unlikely to lead to permanent loss of hearing. A hearing conservation program can become quite complex and expensive if there is not an adequate method for selecting those workers having significant noise exposures.

After a review of available methods for evaluating noise exposures, Wilkening¹ proposed a damage risk criteria (DRC) based upon a combination of the Kryter 2.3.4 and Hardy⁵ recommendations. These criteria have been followed for the past seven years in appraising noise exposures in affiliates of Standard Oil Company (N.J.). Minor modifications, largely for purposes of convenience, have been made but the essential features have remained unchanged. As used recently, the criteria consist of three zones illustrated in Figure 1.

The form shown in Figure 1 is used for evaluating forty-hour per week exposures to broad band noise. Octave band data from environmental noise surveys are first plotted. Sound spectra which fall below the lower border of the light grey zone are judged to be of no risk. On the other hand, plots which penetrate above the upper limit of the light grey zone indicate a high risk of permanent hearing loss with prolonged exposure. The octave band spectra which fall within the light grey zone are considered to be of an uncertain or borderline risk.

Our experience with this method of presenting noise data has generally been favorable. The three-valued concept has been very helpful in emphasizing to management that prediction of hearing loss is a very uncertain undertaking. In the case of "high risk" noise exposure or exposures to insignificant noise levels the course of action is very clear cut. In the case of borderline exposures, however, a number of additional factors must be taken into account. In those cases where part-time or intermittent noise exposures are involved, the evaluation and rec-

ommendations become even more involved and arbitrary. It was because of this that we sought a more workable and easily understood set of criteria for these intermittent exposures.

#### **Hearing Conservation Criteria**

One of the modifications which has been made in the method of rating noise exposures is a change from a damage risk to a hearing conservation criteria (HCC) basis. The use of a damage risk criteria implies that excessive noise exposures will result in a definite degree of hearing loss. On the other hand, hearing conservation criteria indicate a level of noise exposure above which hearing conservation measures become desirable. The later concept is more consistent with the industrial hygiene practice of using Maximum Allowable Concentration values to indicate when corrective action should be taken. The new method is helpful in making more clear cut and consistent industrial hygiene recommendations to management.

The first step in establishing hearing conservation criteria consisted of establishing a guideline for forty-hour-per-week exposures. Basically the lower limit of the borderline risk zone (Figure 1) was accepted as a hearing conservation criterion. Several minor modifications were then made to attain better agreement with the recommendations of the American Academy of Ophthamology

#### DAMAGE RISK CRITERIA FOR NOISE EXPOSURES

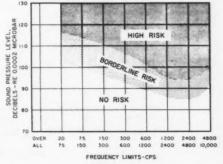


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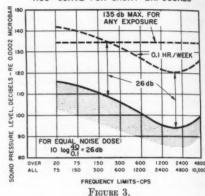
and Otolaryngology<sup>7</sup> and Air Force Regulation 160-3<sup>8</sup> for the 4th, 5th, and 6th octave bands. The value for the first octave was also lowered slightly to coincide with that of Kryter. The two curves are compared in Figure 2.

As was true for the earlier damage risk criteria the new standard applies to a forty-hour-perweek exposure to broad band types of noise.

### Part-time Noise Exposures

In the petroleum industry, and elsewhere, it has been found that many noise exposures are for much less than eight hours per day. For this reason, the DRC had to be interpreted in the light of these shorter exposures. This interpretation usually involved the concept of equal energy which assumes that if the exposure time

HCC CURVE FOR SHORT EXPOSURES





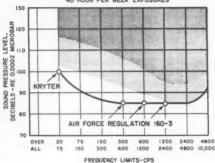
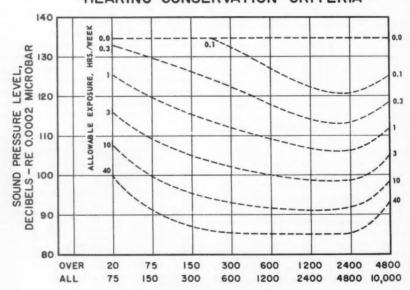


FIGURE 2.

#### HEARING CONSERVATION CRITERIA



FREQUENCY LIMITS-CPS

FIGURE 4.

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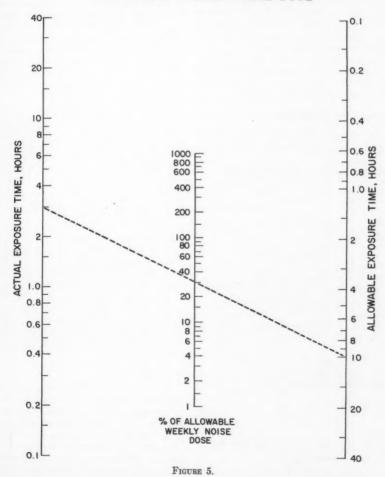
in

is cut in half then the allowable noise intensity could be doubled with no increase in risk. Although the concept of equal energy is convenient, it is too conservative to apply to very short exposure times. A less conservative but realistic procedure has therefore been adopted. The rationale for using this approach is based on some recovery of the temporary threshold shift during unexposed periods. Referring to the original DRC shown in Figure 1, the upper limit of the borderline zone represents an optimistic appraisal criterion for forty-hour-per-week exposures. In order to compensate for the overly

cautious equal energy approach, this upper line has been used in the appraisal of short time noise exposures. The shortest exposure time that has been considered is 0.1 hour per week. The method of establishing the 0.1 hour per week HCC curve is shown in Figure 3.

From this graph it may be seen that the upper limit of the borderline zone has been adjusted upward 26 db. This means that based on the equal energy principle, a noise 26 db more intense can be tolerated at the same degree of risk if the exposure time is reduced from forty hours to 0.1 hour per week. As has been suggested by

## CHART FOR CALCULATING ALLOWABLE WEEKLY NOISE DOSE



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Allen° an additional restraint of 135 db maximum has been adopted for any noise exposure, however short. Thus, the allowable level for the first three octave bands is 135 db rather than the higher values that would be calculated with the equal energy assumption.

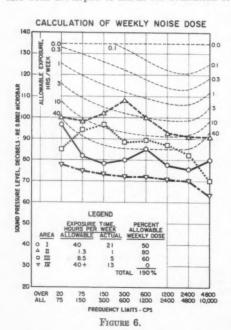
#### Noise Plotting Form

The two preceding sections present curves for evaluating forty-hour and 0.1-hour per week noise exposures. It is now a simple matter to interpolate between these two limits for intermediate noise exposures. A logarithmic interpolation was made between the forty-hour and 0.1-hour per week curves to obtain the intermediate points for the HCC plotting form shown as Figure 4. The new form is used in much the same way as the old one. Octave band noise data are first plotted. The lowest value of "Allowable Exposure" reached by the graph in any octave is taken as the recommended maximum.

For convenience in display, the parameter values follow an approximately logarithmic scale. This fact should be kept in mind when making an interpolation between the curves.

#### Appraisal of Total Noise Exposure

A method of estimating weekly noise dose has also been developed to aid in the evaluation of



non-steady state noise exposures. For each worker, octave band plots are made of the noise spectra to which he is exposed. Those spectra falling below the forty hour "Allowable Exposure" line are not considered to be significant. The "Allowable Exposure" times are then read from the plotting form. The values thus obtained are compared with the actual exposure time of the worker by the relationship:

Per cent of Allowable Weekly Noise Dose

 $= \frac{\text{Actual Exposure Time}}{\text{Allowable Exposure Time}} \times 100$ 

The comparison between actual and allowable exposure times can also be made by the alignment chart shown in Figure 5. A straight line is drawn between the Allowable Exposure Time value and the Actual Exposure Time as determined by observation. The noise dose received by the employee for this fraction of his total noise exposure is indicated on the center scale as "Per cent of Allowable Weekly Noise Dose". This method of calculation is then repeated for each significant noise exposure of the worker. The percentage values thus obtained are added and if the sum is less than 100%, the worker's exposure is considered to be satisfactory. On the other hand if the total Allowable Weekly Noise Dose is above 100%, then there is need for hearing conservation measures to be taken.

The general method of calculating the weekly noise dose is illustrated by the example shown in Figure 6. In this example it has been assumed that the worker spends time in four different areas each having the noise spectra shown in the graph. For each of the spectra, the value for allowable exposure time is read from the plotting form. These allowable exposure time values are then compared to the actual exposure times with the resulting allowable weekly noise dose values shown in the tabulation. This method of plotting and evaluating environmental noise survey results also indicates where corrective action could most advantageously be taken. This is possible because the relative contributions of sound intensity and exposure time can be determined easily.

#### Summary

A new method of appraising noise exposures has been developed. It is based on the concept of hearing conservation rather than of damage risk. Hearing conservation criteria are used to judge whether or not hearing conservation measures should be taken rather than to predict when hearing loss will occur. The method proposed for evaluating noise data takes into account the

more restrictive recommendations made recently for the 4th, 5th and 6th octave bands. Exposure time variations are taken into account by means of a new form that has been devised for plotting octave band measurements. A supplemental graph is presented and can be used for summing up noise exposures at different intensity levels, with the over-all exposure expressed as "Per cent of Allowable Weekly Noise Dose."

#### References

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#### RECRUITING FOR OCCUPATIONAL HEALTH

THE DIVISION OF OCCUPATIONAL HEALTH, U. S. Public Health Service is undergoing considerable expansion and emphasis of new activities following the recent reorganization of the Division and a notable increase in budget. This has resulted in the opening of a number of positions for technical personnel at grades ranging from junior positions requiring no experience up to senior positions at the levels of Section and Branch chiefs. Physicians, immunochemists, pharmacologists, engineers, physiologists, analytical chemists, industrial hygienists, nurses, medical technicians and other disciplines are needed. This great variety emphasizes the broad aspects and opportunities in occupational health today.

The Division has released a brochure describing the various positions for which active recruitment is under way and listing others to be anticipated for the near future. Openings exist in all three branches: Research and Technical Services Branch, Training Branch (including the Technical Information Section), and the States Services Branch. The States Services Branch operates out of the Division headquarters in Washington, D. C., whereas the other two branches are located at the Occupational Health Research and Training Facility, 1014 Broadway, Cincinnati, Ohio.

This revitalization and expansion of occupational health activities at the federal level, with the resulting career opportunities, is welcome news to all industrial hygienists. It is material recognition of the growing importance and stature of the field of industrial hygiene, which reflects to the credit and interest of industrial hygienists in general.

## **Evaluation of Exposures to Industrial Noise**

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DURING the last five or ten years a large amount of data has been gathered concerning the noise levels in industry. Much of this information is meaningless because it represents merely "meter readings", and it does not contain the essentials of noise surveys. This paper will be devoted to discussing the general type of information which must be gathered during a noise survey, the type of equipment to be used, and some of the pitfalls which are encountered while making noise measurements.

First of all, it is assumed that the noise survey will be made in order to evaluate the hazard of a noisy environment causing permanent hearing impairment in those exposed. While noise problems dealing with speech communication, annoyance, neighborhood problems, or engineering surveys for reducing the noise of a particular piece of equipment are of interest and importance to many people, they are beyond the scope of this paper.

#### General Information

The information in the report should be complete and accurate. The name and location of the plant, the date of the survey, the name of the department, the total number of employees in the plant, and the number of employees in each department being surveyed should be included. The number of people who are directly exposed to the noise source should be reported as well as the number who are indirectly or incidentally exposed due to reverberation or to their intermittent presence in the area.

The name, type or model and serial numbers of the noise measuring equipment being used should be listed. The importance of this item is obvious if more than one set of noise measuring equipment is available. It may well be that data taken with a particular set of instruments will be reviewed at a later date if subsequently something of a suspicious nature is found in the per-

formance of the instruments.

When the measurements are being made at particular machines or operations, it is well to

know something about the operating conditions of the noise source in terms of speed. This could be either in parts per minute, strokes per minute, feet per minute, a percentage of its rated capacity or its power requirements. It should be noted whether any other equipment in the immediate vicinity is in operation. Again, if other measurements are made at a later date, it will be possible to compare the actual operating conditions to determine whether any differences in sound levels might be due to operating condiIn eq

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A general description of the construction of the room from an acoustical point of view should be recorded. This enables one to better visualize the conditions. Are the walls and ceiling constructed of brick and glass, concrete, wood, or metal? What is the ceiling height? Has acoustical absorption material been placed on any of the walls or ceilings? Have there been any attempts at noise control in the department? If so, they should be described and opinions recorded as to whether these measures were effective.

A rough sketch of the department showing the approximate dimensions, the locations of machines, the work stations of employees, and the location at which the noise measurements were made is most helpful in visualizing the plant conditions. This sketch becomes useful when comparing measurements made at different dates. It is possible to see readily whether the measuring locations were the same, or whether there were major changes in the plant layout. Actually, much of the information can be recorded in rather brief notes on the sketch.

The time pattern of exposure is an important consideration, and the following type of information is needed to evaluate the hazard. How long is the employee exposed to noise, and what is the time pattern? Is he exposed steadily for eight hours per day for five days a week; is he exposed for only one day per week for eight hours; or is he exposed two hours per day for four days per week? Does he alternate throughout the day at five-minute intervals between high noise-level areas and quiet areas? All of this information enables one to better evaluate the damage risk. While there may be some disagreement about the

Presented at Twenty-first Annual Meeting of American Industrial Hygiene Association, April 27, 1960, Rochester, New York.

equal energy concept, there must be some differentiation in the degree of hazard between one hour per day exposure and an eight hour per day exposure to the same noise. It is also important to know the noise levels to which the employee is exposed when he is away from the more severe noise levels.

Is ear protection worn? If so, by whom and

what type? Plugs or muffs?

Is there an audiometric examination program? If so, when are the examinations performed? Is it a pre-placement audiometric program or is it a periodic examination program? Under what conditions are the hearing tests made? The testing facilities and the environment should be described. The qualifications of the persons giving the tests should be listed.

Are there any unusual conditions, such as a high average age level or extremely long periods of service in noisy departments? Are there any

claims pending?

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The discussion of this supplementary, but very necessary, information has been placed first in order to emphasize its importance. Octave band levels without this additional auxiliary information are almost without meaning. The manner in which employees are exposed, as well as the nature of their acoustical environment must be known if we are to properly evaluate the possibility of damage to hearing.

#### Selection of Equipment

There are several types of noise measuring equipment, each of which has special application, and in each noise situation there is probably one preferred type of equipment. The various situations where a condenser microphone system, a magnetic tape recorder, or an impact noise analyzer should be used will not be discussed here. The characteristics and applications of each of the instruments can be found in the references listed in the bibliography.

For most industrial applications the sound level meter (equipped with a crystal or dynamic microphone) and the octave band analyzer are the instruments which combine to provide good results. The combination with a crystal microphone has been chosen for the example here. The manufacturer provides an instruction booklet for each of the various instruments and the person conducting the noise survey should be familiar with the contents of these booklets.

If the plant to be surveyed is large and the available time is limited, some useful information can be obtained by using a sound survey meter or sound level meter. The noise levels on the three weighting networks (A, B and C),

should be recorded. By using these three values and referring to a family of curves, it is possible to get some idea of frequency distribution in three broad bands. This method, which was developed by J. R. Cox, is described in the 1954 and 1958 editions of Handbook of Noise Measurements, General Radio Company.

#### **Laboratory Check-out**

Before leaving the office or laboratory, the noise measuring equipment should be assembled and checked, in order to insure its proper operating condition. It should be connected and operated in the same manner as in the field. The equipment should be turned on and allowed to warm up for a few minutes. The batteries should be checked. The instruments should be calibrated both electrically and acoustically.

If any of these steps reveal faulty operation, it is sometimes easy to correct the trouble. If the batteries are low, they should be replaced. If the batteries are satisfactory, but the electrical calibration produces no meter reading, then the tubes should be removed and tested. If the electrical calibration is satisfactory, but the acoustical calibration produces no reading, then one must look for trouble in the microphone socket. The inspection might reveal a loose connection or a broken lead.

One could have more serious trouble, such as a faulty microphone. Disconnect the microphone from the sound level meter, and touch each terminal in the microphone socket with a bare wire, pin or paper clip. If only one terminal, the ungrounded one, produces a large reading, it is an indication that the sound level meter itself is functioning and a faulty microphone should be suspected. Such a microphone should be returned to the manufacturer for repair or replacement.

Occasionally, a broken wire or disconnected battery lead is the cause of failure. Such items are usually quickly recognized when the meter is removed from the case. To locate this simple trouble does not require any knowledge of electronics or circuitry, and it is only necessary to be

a good observer.

The octave band analyzer also should be turned on and allowed to warm up. The batteries should be checked. The output of the sound level meter should be connected to the input of the octave band analyzer and the calibrating tones applied to the microphone of the sound level meter. If the overall reading on the sound level meter and the octave band analyzer do not agree, correction should be made on the octave band analyzer by turning the "level" adjustment button. Some oscillator-calibrator sets generate pure tones of

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two different frequencies, and the octave bands containing those tones as well as the overall level should be checked.

As before, the batteries should be replaced if they are low. If there is a response on the sound level meter but not on the octave band analyzer, the patch cord which connects the two instruments may be defective. If this is not the case, the tubes should be removed and tested.

In most field measurements the microphone is used on the end of a cable and the instruments should be checked with the cable in use. Sometimes a wire in the cable becomes broken near the socket due to frequent flexing and bending.

Because equipment of this type can usually be repaired easier and better at home than in the field, it is good practice to thoroughly check the equipment before leaving on a trip.

#### **Travel Precautions**

Some people are so fortunate as to not have to travel with the instruments, but there are those who must transport them around the country. When using public transportation systems, it is best to carry the instruments and not to trust them to baggage handlers. They should not be checked through to your destination.

If the instruments are being transported in an automobile, it is best to place them on the seat or if they are to ride in the trunk, place them on some sort of a resilient pad.

The crystal microphone, which is found in sound level meters, can be damaged if subjected to temperatures of more than 115°F. This temperature often can be induced in the enclosed case by leaving the instrument in the trunk of the car, in the closed passenger compartment or by simply leaving it on the shelf by the rear window. The microphone can be disconnected easily and carried either in a pocket or briefcase to avoid such heat damage.

#### Field Checks

When you arrive in the field, the basic steps of battery checks and acoustical calibration should again be performed. When using a cable with a crystal microphone there is a loss in the cable which is dependent upon the temperature of the microphone. Consequently, it is wise to bring the equipment into the plant prior to the survey, if possible, and allow it to stabilize. There are charts available showing the cable correction due to the temperature. Another procedure is to calibrate the sound level meter first with the microphone attached directly to it; then calibrate it with the microphone on the cable. This difference in readings gives the correction number which must

be added to the meter readings due to the use of the cable. However, if one should forget to do this, a reasonable and practical correction figure seems to be 3 db for a cable length of 25 feet.

#### Selection of Measurement Locations

Selecting the location for the noise measurements requires some thought. One is interested in measuring the employee's exposure to noise but unfortunately, his head occupies the space where the microphone should be placed. The microphone should not be placed within a foot or two of the employee because reflections from his body will give readings greater than the true exposure. Normally one can select a spot two or three feet from the employee which corresponds pretty much to his actual exposure. If the employee works at the center of a machine, stand to one side. If he works at the side of a piece of equipment, stand on the opposite side of it. If a person is holding the microphone (rather than placing it on a stand or tripod), he should hold it at arm's length to reduce the effects of reflections from his own body.

#### **Equipment Operation**

Generally, the noise should strike the microphone at a random incidence. Differences in levels of 5 db or so are observed between grazing incidence and zero degrees at frequencies above 2000 cps. For example, if the microphone is pointed towards the opening of the open end of a tumbling barrel, there will be a much higher reading than if the noise passes at a grazing incidence.

The attenuator on the sound level meter should be adjusted so that the indicating meter reads on scale. When the octave band analyzer is connected to the sound level meter, it should also read on scale for the overall measurement. In reading the levels in the various octave bands, the attenuator on the octave band analyzer is adjusted so that the meter reads on scale. On some analyzers there is an adjustable attenuator dial which is graduated in 10 db steps. When there is zero attenuation on the octave band analyzer, this loose dial should read the same as the attenuator on the sound level meter. Many people perform the arithmetical operations mentally as they adjust the attenuator, but this is a point where errors of 10 db frequently occur.

Because of the possibility of such errors it is always well to check the overall reading by adding up the levels in each of the octave bands. This process, which is described in various texts and references, is not a difficult system to learn. It is accomplished by making a series of seven additions so that the level in one band is combined

with the total level of all bands previously com-

Frequently it is possible to eliminate unimportant bands by starting with the bands with the highest levels. It is not necessary to know all of the numbers shown in Table I. It is necessary to remember only a few numbers in order to check the measurements in the field. For example, if the difference in levels between two octave bands is zero, 3 db is added to the value; if the difference is 2 db, 2 db is added to the higher value; and if the difference is 6 db, one db is added to the higher value. If this calculated value is not within one or two db of the overall reading, it is well to repeat the entire octave band analysis.

Frequently the sound fluctuates by 2 or 3 db and such variations are not difficult to read on the meter. However, when there are rather wide fluctuations, it is difficult to determine the average value. By turning from the fast scale to the slow scale, a good idea of the average value is obtainable. However, it is well to know the entire range of fluctuations. This could be an impact noise of some type that occurs regularly in the operation of the machine. Sometimes such impulse noises will peg the needle at the high end. By adjusting the attenuator 10 db or higher, the swing of the needle is not as great and a more accurate measurement is obtained.

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Vibration of the instruments can produce false readings. If the octave band analyzer produces a reading when the patch cord from the sound level meter has been removed, it is an indication that the instrument is responding to vibration. The octave band analyzer should be raised from the floor or other vibrating surface, and if the reading disappears it is an indication that airborne sound is not being measured. The instruments can be held or placed on a resilient pad to reduce the effect of vibration.

In high level noise fields there may be a meter reading because the tubes are being vibrated, creating microphonics. By using 50 or 100 feet of microphone cable, the instruments can be placed in a quieter location such as a shop office or behind a wall which will provide some shielding. If long lenths (over 50 feet) of cable are to be used, the dynamic microphone system in preferable

Electrical and magnetic fields can also affect the operation of the instruments. This has been noticed in resistance welding departments, in the vicinity of the electric power transformers, in the vicinity of very large electric motors, and in a department where high frequency portable electric tools were being used.

Extreme variations in readings in the lower

Table I
Chart for Combining Noise Levels

Excess of stronger component	Add to stronger for combined level db		
0	3.0		
1	2.5		
2	2.0		
3	1.8		
4	1.5		
5	1.2		
6	1.0		
10	0.5		

frequencies have been disconcerting at times. By monitoring the output of the octave band analyzer, one can sometimes recognize the noises and then look for the source. In one case it was found that the variation was due to the loading and unloading of the air compressor about 150 feet away. It has also been noted when due to the intermittent operation of rather large unit heat-

Earlier, the need for calibrating the equipment before making the survey was mentioned. Checking the calibration during the survey is also important. It is good practice to calibrate every hour and a half or two hours, during a survey and also upon the completion of the survey either at noon or at the end of the day. While it is most disconcerting to find large differences in consecutive calibrations, frequent calibration can save a tremendous amount of work. One always wonders when and where the change occurred. Did the change occur immediately after the previous calibration or just before the most recent calibration? With frequent calibration it may be necessary to question or throw away only data which was accumulated in a short period between calibrations, whereas, if you calibrate only at the beginning of the survey and at the end of the survey, you may have to repeat the entire survey in the interests of reliability.

Winds of greater than a few miles per hour may introduce errors in noise measurement. The air passing by the microphone will generate vortices or whirlpools of air because of the microphone's irregular surface. These vortices can cause fluctuations in atmospheric pressure which actuate the microphone. Usually wind-noise affects only the lowest octave bands but these will flucutate violently. Sometimes variations of 10 to 20 db can be observed as gusts or puffs of wind pass by. These can be detected by listening to the output of either the sound level meter or the octave band analyzer. A wind screen, which is frequently in the form of a cylinder with a fine silk

or nylon stretched over a wire frame, can be used and will prevent these wide fluctuations.

We have discussed some of the information which is needed for a proper evaluation of a noise hazard. We have also discussed some of the more common difficulties encountered while making a survey. There are other specialized pieces of equipment for certain types of noise and they introduce still other measuring techniques and operating difficulties. The impact noise analyzer, the narrow band analyzer and the cathode ray oscilloscope can be better used in the laboratory than in the field. Their use then, necessitates recording the noise on magnetic tape and performing the analysis in the laboratory. If the laboratory is not too far from the noise source, one can return to the plant for further information or retaping the noise without a great deal of inconvenience. However, when the plant is hundreds of miles from the laboratory, it becomes necessary to develop an almost foolproof technique in the use of the tape recorder. For intermittent, transient, impact and impulse noises, the tape recorder has many advantages. However, the method can be troublesome and there is much

to be said for actually doing the frequency analysis in the field whenever possible.

#### Conclusions

There is much more to making a noise survey than reading the dials on a sound level meter. Care must be exercised in gathering and recording the pertinent data relating to the environment, the exposure and the sound levels. In addition, it is essential that the person making the sound level measurements be familiar with the operating characteristics of the equipment, so that warning signs of malfunctioning of the equipment can be recognized. This will help insure reliable measurements in the field, where conditions are frequently far from ideal.

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#### VENTILATION CONFERENCE

THE TENTH ANNUAL INDUSTRIAL VENTILATION CONFERENCE will be held at Michigan State University, East Lansing, Michigan, February 20–23, 1961. This conference is a three and one-half day workshop to present design information and practical solutions for better air handling. Engineers, contractors, and others concerned with good ventilation find the conference a fine meeting place for the exchange of ideas and up-to-date information. Lectures and demonstrations are practical and informal, covering such subjects as hood design, fan selection, dust collectors, make-up air, and air distribution. Classroom problems include design calculations, velocity selections, mine ventilation, dust control, and others.

The sponsors of the Conference are the Division of Occupational Health, Michigan Department of Health; College of Engineering, Michigan State University; and Continuing Education Service, Michigan State University. Inquiries and applications should be addressed to Industrial Ventilation Conference, Continuing Education Service, Michigan State University, East Lansing, Michigan.

## **Evaluation of an Aerosol Photometer** for Dust Counting and Sizing

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#### Introduction

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N 1957 at the Nineteenth Annual Meeting of the American Conference of Governmental Industrial Hygienists, Dr. Lewis J. Cralley summarized quite clearly the need of industrial hygienists for a simpler, less fatiguing, faster, and more accurate technique for evaluating dust exposures. Dr. Cralley stated:

"... However, the tediousness, limitations and lack of uniformity in techniques, along with the time required, deter collecting the mass of data on dust exposures needed for correlation with clinical findings. . . . additional environmental data are needed on the extent, frequency and duration of peak dust exposures, on the relation of particle size distribution to changes in dust concentrations, and on the many factors which influence the characteristics of air-borne dust. Perhaps this will come with the advent of atmospheric dust characterization through automation.... The ideal instrument should sample the air directly so that the air-borne particulates of hygienic significance are automatically counted, sized and recorded as they exist in the air."

Various types of automatic counting and sizing technics for aerosols have been introduced since 1944. An excellent review of these devices and a description of an improved instrument is given by O'Konski et al.2 Fisher et al.3 reported the development of the Aerosoloscope-an instrument for the automatic counting and sizing of aerosol particles. Because of size, weight, or operating limitations, however, most of the instruments which had been developed seem more appropriate for laboratory use than for industrial hygiene field use.

In 1955 this laboratory, still seeking an instrument to reduce the time consumed and the eye fatigue in counting dust, investigated the use of an automatic blood cell counter. Christman and Hosey' concluded that since this particular instrument was designed for counting blood cells of approximately 8 microns ( $\mu$ ) and

since most dust particles of hygienic significance range from about 0.5 to 5  $\mu$ , its sensitivity was inadequate for dust counting. At that time it was believed that this instrument could be modified to count samples collected with the impinger. If, on the other hand, the step of collecting the sample in an impinger could be eliminated it would be possible to evaluate aerosol concentrations as they actually exist in the

At about this same time (1955) our laboratory purchased a light scattering photometer for monitoring dust concentrations in animal exposure chambers. Fraser<sup>5</sup> found this a very useful instrument for that purpose. Again, however, this was not the type of device to be used in the field for evaluating dust concentrations. It was not a portable apparatus, it required a source of electrical power, it was difficult to calibrate, and it had a considerable amount of zero drift.

This report describes an aerosol photometer developed by the Southern Research Institute (SRI)6,7,8 and gives preliminary results of comparative tests made with it and the midget impinger, using the standard light field counting

technic.

An earlier model of the SRI aerosol photometer was made available to our laboratory for preliminary testing in February 1959. Results of these tests indicated that this instrument had definite possibilities for field use. It was small, light-weight, battery operated; it indicated results in millions of particles per cubic foot of air; and it was possible to determine particle size distribution by manually changing a discriminator circuit. In June 1959, a contract was awarded by the Occupational Health Program of the U.S. Public Health Service to the Southern Research Institute to develop and construct a modified version of the earlier model. The present report is based on work done on the modified aerosol photometer, which was received in January 1960.

#### Description of the Instrument

Figures 1 and 2 are photographs showing exterior and interior views of the aerosol photom-

This report does not constitute an endorsement by the Public Health Service of the instrument described for dust counting.

eter (referred to hereinafter as AP). Figures 3 and 4 illustrate the operating principles of the instrument. During operation of the instrument, the sampled aerosol passes through a beam of light. Light scattered from individual particles is sensed by a phototube, the impulses from which pass through amplifying, discriminating, and pulse rate circuits to be indicated as particle concentration on the panel meter.

The components are housed in an aluminum case which measures 5 inches wide by  $8\frac{1}{2}$  inches high by 15 inches long (over-all), and the weight is  $10\frac{1}{2}$  pounds. As shown in Figure 2, one side of the case is hinged to permit easy

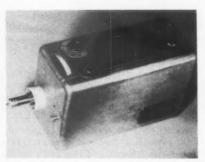


FIGURE 1. The Aerosol Photometer—sampling probe and dilution valve control extend from front of case; indicating meter at center; size selector behind and to right of meter; count and battery check switch behind and to left of meter; and onoff switch on top rear of case.

access to the components. A magnetic catch is provided to keep the case closed tightly and prevent entrance of dust into the interior. Shown at the left in Figures 1 and 2 is the air sample inlet and dilution valve selector. A patented dilution valve is used which permits a range of full-scale readings from 5 to 100 million particles per cubic foot (mppef). Thomas et al.<sup>8</sup> describes the operation of this valve as follows:

"The dilution valve can be used to extend the upper concentration limit by any factor up to 10 or 20. In the dilution valve, clean air is added to the sample aerosol, and the excess is discarded without subjecting the sample to sharp changes in direction that would trap larger particles. The sample air and filtered air are drawn through tubes of equal resistance to a place where they taper into D-shaped openings, the D's facing each other to form a round outside. From similar openings, 2 tubes lead on, one going through the light and another going around the light to the exhaust. The inlet tubes can be rotated with respect to the outlet tubes, resulting in a division of the sample stream between the outlet tubes and a corresponding division of the clean air between the outlet tubes. Thus the ratio of sample to clean air going to the counter can be varied infinitely at will by simple rotation of a single part. With the inlet tubes of equal resistance and the outlet tube of equal resistance, there is no change in any flow rate as the valve is rotated. The filter from which dilution air is obtained must be of very low resistance for this to hold true."

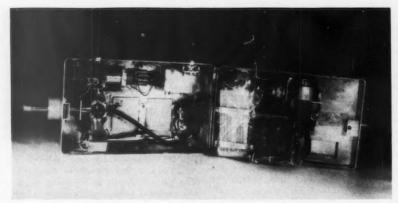


FIGURE 2. Interior view of photometer showing optical system, counting chamber and phototube at left; dilution air filters at center; high voltage supply upper right and below this the lamp and recorder rheostats; recorder connectors and charger plug-in at the rear center; rechargeable batteries and mercury cells, motor and gear pump, and electronic circuit boards are shown on the hinged door lid.

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A 1:1 dilution means one volume of sample to one volume through the sensing chamber (i.e., no dilution); 20:1 would be one volume of sample per 19 volumes of dilution air so the sample is 1/20 of the mixture in the sensing chamber.

Thomas further states, "Clean air is also admitted to the body of the photometer, preventing the sample from accumulating in the chamber."

A small gear pump, driven by a 4-volt, d.c. motor (equipped with a governor) supplies the suction required. Air is sampled at 17 cubic centimeters per minute in the present model. The rate of air flow is rather critical and must be checked periodically. This is done by the "soap bubble" technic using a 25 or 50 ml

burette and stopwatch.

The optical system is shown schematically in Figure 4 and at the left of the interior view, Figure 2. A General Electric PR-3 prefocused flashlight bulb, operated at about 3 volts, was used originally as the light source but a specially designed type of bulb was installed after most of our tests were completed. An RCA 1 P 21 phototube, operated at about 950 volts, picks up light pulses from particles passing through the light beam and produces a current for each pulse of light. Output from the phototube is fed into an amplifier to increase the height of the pulses so they can be handled by the remaining circuits. The amplifier output is fed into an amplitude discriminator, which can be manually adjusted to accept voltages of various values depending upon the particle size of the aerosol under test. Finally, the output of the discriminator circuit is fed into a log pulse rate circuit which gives an output voltage proportional to the number of particles per unit of time. This voltage is indicated on a logarithmic scale, graduated from 0.01 to 5 mppcf full-scale, by the panel meter shown at the top left in Figures 1 and 2

Power for the motor, light source, and high-voltage supply is furnished by 3 Nife type C-5.9M, 1.35 volt nickel-cadmium rechargeable cells. Two, 8-volt, Mallory TR-136 mercury cells supply the necessary "B" voltages. The two mercury cells were replaced after three months of more than normal usage. Life of the wet cells is expected to be over a year.

The discriminator circuit can be adjusted manually in 0.1  $\mu$  increments to count only particles larger than a pre-selected size within a range of from 0.3  $\mu$  to 2  $\mu$ . Although the instrument will count particles larger than 2  $\mu$ , no size discrimination is possible above this limit with this particular instrument.

Calibration of the discriminator circuit or cut-off points can be accomplished by sampling

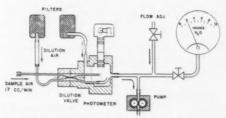


FIGURE 3. Schematic diagram of air flow system. (Drawing reproduced by permission from Southern Research Institute.)

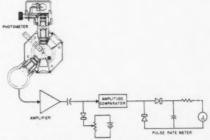


Figure 4. Schematic diagram of optical system. (Drawing reproduced by permission from Southern Research Institute.)

an aerosol of known size. Polystyrene latex spheres are convenient for this purpose and can be obtained from the Dow Chemical Company in various measured sizes from about 0.1  $\mu$  to 2 μ with extremely narrow ranges for each stated size. A calibration curve with the discriminator voltage plotted against particle size was supplied with the instrument. The curve was checked in this laboratory using 0.365  $\mu$ , 0.554  $\mu$ , 0.814  $\mu$ , 1.17  $\mu$ , and 1.8  $\mu$  polystyrene latex spheres and the calibration equipment supplied with the instrument, which included a nebulizer and a small chamber made from a pint paint can with an "absolute filter" attached to the air inlet side. An oscilloscope was attached to the signal jack provided with the instrument. Particle size was characterized by the pulse heights appearing on the scope. Also, by slowly adjusting the discriminator selector the cutoff point, when pulses no longer trigger the discriminator circuit. can be determined. A few words of caution should be interjected at this point. Dust-free distilled water must be used to dilute the concentrated latex suspension for use in the nebulizer. Either double distilled or distilled water which has been passed through a membrane filter should be used. Furthermore, air filtered through an absolute filter must be used for the squeeze-bulb of the nebulizer. If these precautions are not observed, spurious counts will be obtained. If the concentrated latex suspension has settled or agglomerated after long standing an ultrasonic generator can be used to redis-

perse the spheres.

A full-scale reading at 1:1 dilution indicates a concentration of 5 mppcf and the lowest detectable concentration is 10,000 particles per cubic foot. For this particular instrument, it can be determined mathematically that 50 pulses per second at a flow-rate of 17 cc per minute will indicate 5 mppcf. A pulse generator is connected across the amplifier input and set for 50 pulses per second at 0.1 volt output. With the discriminator circuit set at one  $\mu$  the "trim pot" on the pulse rate circuit is adjusted to give a full-scale reading of 5. The relationship between pulse rate, aerosol concentration, and flow is:

 $Flow = \frac{pulse\ rate}{concentration}$ 

For example, flow = 50 pulses/sec  $\times$  60 sec/min  $\times$  28.3 liters/cu. ft.  $\times$  1000 cc/liter  $\div$  5,000,000 particles/cu. ft. = 17 cc/min. The actual air-borne aerosol concentration is the dial reading times the dilution ratio. Thus, at a 20:1 dilution the maximum concentration determined with this instrument is 100 mppcf.

#### **Test Procedures**

Threshold Limit Values (TLV) for pneumoconiosis-producing and nuisance dusts are expressed in terms of the standard light field counting technic proposed by Greenburg and Bloomfield<sup>10</sup> in 1932 and adopted as a standard technique by the American Conference of Governmental Industrial Hygienists<sup>11</sup> in 1942. The test procedure to be described is designed to compare the aerosol photometer (AP) with the impinger sampling and counting technic since the TLV's and many state regulations are based upon this latter procedure.

Tests were conducted in the laboratory using a single, fairly "pure" dust for each series of tests. In our opinion, this was more appropriate than field tests for initial evaluation since the concentrations could be more uniformly controlled and a number of variables could be eliminated. Field tests will be made at a later

date

Utilizing the experience gained by Fraser and co-workers<sup>12</sup> in animal exposure experiments, the laboratory room air flowing through the test chamber was passed through an electrostatic

precipitator to reduce the background dust. A Wright dust feed apparatus was used to disseminate the various test dusts.

The following test procedure was used to determine the cleanliness of the air entering the test chamber: Laboratory air was passed through a Westinghouse electrostatic precipitator 21 inches by 35.5 inches by 8 inches with parallel plates charged negatively with two positively charged wires located at the entrance. The precipitator was operated at about 18,000 volts, and the clean air flowed into a chamber of 125 cubic feet capacity at a rate of about 100 cfm. The AP (set to count  $0.6 \mu$  and above), and a membrane filter holder were attached to the chamber and simultaneous samples were collected. The membrane filter was counted using light field illumination with a 10x objective and the result was 15,000 particles per cubic foot. The AP also indicated an average count of 15,000 particles per cubic foot during the collection of the membrane filter sample. Thus the air entering the chamber was relatively free of detectable particles. It is interesting to note that even at this low concentration the AP indicated a count comparable to the membrane

The Wright dust feed apparatus<sup>12</sup> was placed so as to discharge the dust into a 3-inch diameter pipe connecting the blower to the chamber. Adjustment of two valves in the piping system permitted a portion of this air to bypass the chamber thus affording a method of controlling the dust concentration inside the chamber in addition to the control afforded by varying the amount of dust disseminated by the Wright dust feeder. For example, if the Wright dust feeder were set to give a concentration of 25 mppef with 100 cfm of air flowing through the chamber, the concentration could be nearly doubled by reducing the amount of clean air entering the chamber by a half.

The chief difficulty in establishing various dust concentrations was encountered in packing the dust into the feeder cylinder. Of all the test dusts, natural diatomaceous earth was the most difficult to disseminate. This was probably due to the porous nature and electrostatic properties of the material. Satisfactory dissemination was finally achieved by moistening the diatomaceous earth with distilled water, packing it into the Wright dust feed cylinder by means of a hydraulic press, and drying the resultant mass overnight in an oven at 40°C. With the other dusts used in these experiments, satisfactory results were obtained by packing the dry material into the cylinder at pressures varying from 1,000 to 3,000 pounds per square inch. The compressed air supplied to the Wright dust feed was varied from 2 to 5 pounds per square inch depending upon the particular material used.

Previous experience with this particular chamber, which has several sampling ports located on three sides, showed that a rather uniform concentration of dust could be maintained if an electric fan were placed inside the chamber. Of course, there was some settling of the larger-sized dust, especially at low air flow through the chamber; but the impinger and membrane filter samples contained dust up to 10 microns in size.

The AP with attached 10 millivolt recorder and a commercially available light scattering photometer were located on one side of the chamber while impinger and membrane filter samplers were located on the opposite side. The recorder attached to the AP provided a permanent record of the dust concentration and indicated the range of concentration during the time other samples were collected. Visual estimation of the record made during a test was used to obtain the average concentration for a particular run. The other light scattering photometer, while not calibrated in terms of dust concentrations, indicated whether the chamber dust loading was uniform or variable during a run and served as a rough check on changes in concentration when the air flow or Wright dust feeder was altered.

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Initial efforts to sample with the aerosol photometer directly from the dust chamber were unsuccessful. A positive gage pressure of about ½ inch of water existed in the chamber; this forced air through the instrument and disturbed the dilution valve ratio. Under these conditions the dilution valve did not function properly and high counts were indicated. A special sampling port was devised to overcome this difficulty. The end-opening in one long leg of a small glass tee connection was further restricted by flaming. The opposite end was also restricted but to a lesser degree. This end was placed in the chamber. No change was made in the side arm, which served as a bleeder valve. A one-inch length of rubber tubing was attached to the less restricted opening and the instrument probe was inserted into this rubber tube. This was not a close fit because it was necessary for the excess air forced out of the chamber through the tee to bleed off around the sampling probe. To learn whether this device might alter the sample, the AP was placed inside the chamber and operated for 15 minutes. The AP was removed from the chamber, attached to the glass sampling tee, and was operated for about 15 minutes. The average concentration indicated by the re-

corder was essentially the same during tests inside and outside the chamber.

In actual operation, the Wright dust feeder was loaded with the particular dust under test; the rate of air flow and dust feed was adjusted to give approximately the desired concentration; and the system was operated for a period of 15 to 30 minutes to reach equilibrium as observed by the monitoring photometer. Impinger samples, both midget and Greenburg-Smith on some tests, were collected for 5, 10 or 15 minutes depending upon the concentration. In earlier tests membrane filter samples were collected at 0.1 cfm for 15 to 60 seconds during the 5 to 15 minute impinger sampling time. In later tests membrane filter samples were collected at 1/2 liter per minute for one to four minutes near the midpoint of the companion impinger samples. This change in procedure resulted in more comparable impinger and membrane filter samples because the concentration during the earlier brief membrane filter sample was seldom the same as the average concentration for the 5 to 15 minute impinger sample.

Impinger samples were counted in Dunn cells using a 30 minute settling time, light field illumination and a binocular microscope at 100× (16 mm objective and 10× oculars). Duplicate Dunn cells of each impinger sample were counted by two and sometimes three persons. While average values only are shown in the tables and graphs, in the majority of cases two or three persons experienced in dust counting were able to check each other within about 15% on the same sample.

One author perferred to use phase contrast for counting dust in the Dunn cells on certain samples such as the diatomaceous earths and silica flour. The other two authors could check these same samples using light field illumination. Membrane filter samples of silica flower, flux calcined diatomaceous earth, and other dusts presented counting problems which will be discussed in a separate publication. For purposes of this paper, the membrane filter samples were collected to correlate particle size distribution with the AP. For particle size determinations a small circle of the filter was cut out with a No. 4 cork borer (0.3 inch) and placed dust side up on a microscope slide. A drop of immersion oil was placed on the filter and a cover slip placed on top of the filter. Oil was also added on top of the condenser and on the cover slip. Two hundred particles were sized using a Porton eye-piece graticule. Magnification of the optical system was about 1000× and under these conditions the smallest circle on the graticule represented 0.48  $\mu$  and the largest circle 5.44  $\mu$ . The

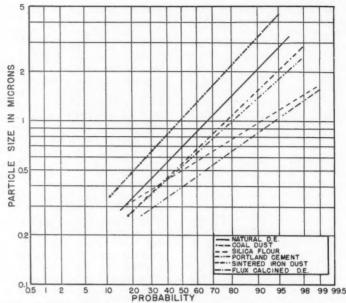


FIGURE 5. Particle size distribution of original bulk materials tested. Coal dust was passed through a 325 mesh sieve prior to use in tests.

oil used consisted of approximately three parts light mineral oil and one part alpha chloronaphthalene. To such a mixture small increments of one component were added until the mixture had a refractive index of 1.507, the value recommended by Crossman.<sup>13</sup> With the particular batch of membrane filters used, an oil with this refractive index was found to clear the filter and leave it almost free of any grainy structure when observed under the optical microscope. Membrane filters from another manufacturer may require the use of an oil with a slightly different index of refraction.

For samples of dust having refractive indices nearly the same as the oil used to clear the filter a modification of the technique described by Yaffe, et al. was used. A small circle of membrane filter containing the dust was placed on a microscope slide dust side up. This was held in place by a gummed, cloth, reinforcing ring for a notebook. Using a micropipette, 0.01 ml redistilled acetone was placed on the filter to clear it. Unfortunately, a sample prepared in this manner cannot be examined under oil immersion but an approximate size distribution can be determined by viewing the sample under a 97× objective used dry. Naturally the resolution under these circumstances leaves much to be desired.

#### Results

Particle size analyses were made on the test dusts. Coal dust was obtained from rafter samples in a tipple and was passed through a 325 mesh sieve prior to use. All other dusts except the sintered iron were manufactured products and used as received. Results are shown in Table I and graphically in Figure 5.

Data on comparative tests with the aerosol photometer and impinger are shown in the Tables II, III and IV and Figures 6 and 7. Tables II and III are inserted to illustrate the type of data collected and the methods used to prepare the summary information shown in Table IV.

Table I
Optical Size Analysis of the Various Test Dusts

	Particle size—per cent less than stated size in microns					Geom. mean mi-	Std. Geom	
	.48	.68	.96	1.36	1.92	2.72	crons	Dev.
Natural D.E	31	56	67.5	77.5	84	89	.7	2.54
Flux calcined D.E	10	33	50	64	73	81	1.0	2.8
Portland cement	45	67	79.5	86.5	91.5	94.5	.54	2.6
Silica flour	46	75.5	90	95	98	99	.5	1.84
Coal	40	65.5	74.5	86	93.5	96.5	.54	2.52
Sintered iron	62	83	93	97	98	99.5	.4	1.95

TABLE II

the Aerosol Photometer and Midget Impinger

AP average Conc. (mppcf)*	Midget impinger Conc. (mppcf)†		MI average Conc. (mppcf)	Ratio AP/MI	
36	29	32.2	30.6	1.2	
7.5	8.9	12.7	10.8	0.7	
15.5	13	17.4	15.2	1.0	
9	11	19	15	0.6	
12.5	19.5	24	21	0.6	
44	104	125	114.5	0.4	
18	13.4	12.5	12.9	1.4	
14	9.6	_	9.6	1.5	
15.5	17.5	11.0	14.3	1.1	
14	8.8	6.3	7.5	1.9	
14.3	11.2	11.8	11.5	1.2	
86	185P	156	170	0.5	
94	167P	162	164	0.6	
83	67P	76	72	1.1	
26	32.2P	24.2	28	0.9	
34	34P	37.7	35.8	0.9	
40	29P	30.6	30	1.3	
44	24P	28	26	1.7	

1.0 Median

\* Discriminator set to count particles ≥0.75 µ. † Duplicate Dunn cells from same sample counted by two individuals.

P = Phase contrast, others light field.

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TABLE III

Comparative Results on Flux Calcined Diatomaceous Earth Sampling with the Aerosol Photometer and Midget Impinger

AP average Conc. (mppcf)*	Midget impinger Conc. (mppcf)†		MI average Conc. (mppcf)	Ratio AP/MI
6	30P	27	28	0.2
28	36P	37	36.5	0.8
68	47P	68.5	57.8	1.1
19	16P	29.4	17.7	1.1
9	15P	16.2	15.6	0.6
23	21.3P	30.2	25.7	0.9
50	43.5 <sup>P</sup>	72.2	57.8	0.9
7	14.1P	12.8	13.5	0.5
5.7	7.2P	9.6	8.4	0.7
7.2	13.6P	14.1	13.8	0.5
15	20.2P	22.9	21.5	0.7
14.5		14.1	14.1	1.0
12.5	11.4P	12.8	12.1	1.0

Median .8

\* Discriminator set to count particles  $\geq 0.6 \,\mu$ . † Duplicate Dunn cells from same sample counted by two individuals.

.77

P = Phase contrast, others light field.

Average

TABLE IV

Comparative Results on Coal Dust Sampling with Comparative Results on Various Dusts-Summary of All Tests. Median Values Based on 7 to 18 Samples for Each Dust

Material	AP/MI at 1 μ*	AP/MI at .75 μ*	AP/MI at .6 μ*
Natural D.E.	1.3	2.1	_
Flux calcined D.E	0.3	-	0.8
Silica flour	0.5	0.9	_
Cement	1.1	-	4.8
Coal	0.6	1.0	-
Sintered iron	1.0	-	3.3

\* Discriminator set to count particles of stated size and larger.

TABLE V Calculation of Particle Size Distribution from Aerosol Photometer Data

Discriminator setting = or > stated size	No. particles (mppcf)	Cumulative No. = or < stated size*	Cumulative per
$0.35\mu$	90		
0.4	60	30	33.3
0.5	29	61	67.8
0.6	19.5	70.5	78.3
0.75	12.5	77.5	86.0
1.0	7.5	82.5	91.7
1.25	4.7	85.3	94.7
1.5	2.5	87.5	97.2

\* Since the AP counts are = or > the stated size, it is necessary to subtract the number of particles at the larger size from the number at the smaller size to obtain the cumulative number at a size = or < the stated size. Thus, 90 - 60 = 30; 90 - 29 = 61(the first two numbers in column 3); etc. The cumulative percentage is obtained by dividing the figures in column 3 by 90, which was the total number of particles counted with the discriminator set to count all particles ≥ 0.3 µ.

Figure 6 is a graphical presentation of the data from Tables II and III respectively. Average impinger counts are plotted against the corresponding average aerosol photometer readings on 3cycle log-log paper. These figures are included to show the spread in results.

The example shown in Table V illustrates one method for determining the particle size distribution from data obtained with the aerosol photometry. The data in column 4 can then be plotted on log probability paper. This is shown graphically in Figure 7. Also shown, for purposes of comparison, is an optical size analysis made on a membrane filter sample of the same material (coal).

Another and perhaps more useful portraval of particle size distribution is obtained by plotting on 3 cycle log-log graph paper the number of particles against the size in microns (= or >). Plots of the data from columns 1 and 2 of the preced-

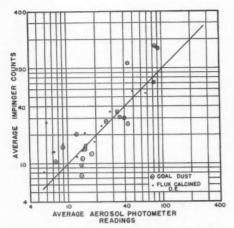


FIGURE 6. Comparison of photometer readings with impinger counts. When the AP was set to count above 0.75  $\mu$  for coal dust the median ratio AP/MI was 1.0. On flux calcined diatomaceous earth with the AP set to count above 0.6  $\mu$  the median value was 0.8.

ing table and similar data from a previous test are thus shown in Figure 8.

#### Discussion

#### Aerosol Photometer Performance

Electrically, the instrument performed satisfactorily during the six months it has been used with the exception of a high-voltage supply failure and the failure of one transistor in the amplifier circuit. These components were replaced and normal performance was restored. The original calibration curve was checked a number of times during these tests and the cut-off points remained the same. The air sampling rate was quite uniform provided the battery was fully charged. It was necessary to adjust the speed of the gear pump motor only infrequently to maintain the desired sampling rate of 17 cc per minute. The two dilution air filters supplied with the instrument performed satisfactorily throughout these tests.

Within the first month of operation the dilution valve failed to operate properly. Rough edges or burrs on the valve face had cut small bits from the rubber O-ring seal and these plugged the valve opening. After the valve faces were polished to remove the burrs it operated more smoothly and, except for periodic cleaning, it gave no further trouble.

If the instrument is used in very dusty atmos-

pheres frequent cleaning of the valve may be necessary. No completely satisfactory simple method has been found, but compressed air was used with success in some instances, while in others it was necessary to use a small swab followed by the use of compressed air.

One of the most troublesome and vexing tasks was replacing the light source. Originally the light source, rated at 3 volts, was operated at 3.3 volts since the AP was so calibrated. This lamp was replaced twice during two months of intermittent operation; once because it burned out and the second time because it became blackened which reduced the light output. Each time a lamp was replaced the optical system had to be refocused and a calibration check made with polystyrene spheres. After much manipulation of the light source and optical system, it became possible to operate the lamp at about 3 volts and obtain the same light intensity as at 3.3 volts. After this change was made the lamp failed after one month's operation of about 3 to 5 hours per day. A new lamp lasted for another month under these same conditions. The data on the first five dust samples reported in this paper were obtained with this original type of lamp.

In May 1960 the GE PR3 lamp bulb was replaced by a special type of short filament bulb operated at 3 volts. This new lamp has an estimated life of about 5,000 hours which should eliminate nearly all of the objections and difficulties encountered with the original lamp.

Use of the new type of light source in future models of this instrument is recommended. Maintenance difficulties would be reduced if provision were made for easy access to the optical system for changing lamp bulbs and refocusing. Changes in the dilution valve assembly to permit easier cleaning and to provide more space between the 5:1, 10:1 and 20:1 settings would be desirable.

#### Dust Counts

Referring to column 3 of Table II, and remembering that two experienced persons made these counts on coal dust, which is quite visible, the range of differences in their counts is from 3 to 42% with a median value of 14%. Treating the data from column 3, Table III, in the same manner, the range of differences is from less than 3% to nearly 46% with a median value of 11.5%.

Variations of about 10 or 15% on 12 to 18 different impinger samples counted at different concentrations is to be expected. The concentrations indicated by the aerosol photometer, for the various dusts tested during this project, fall generally within this same variation. Table IV shows that four of the six dusts tested were within these

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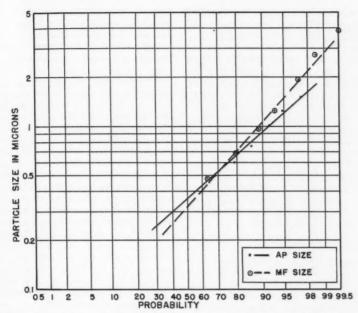


FIGURE 7. Comparison of particle size distribution on coal dust by aerosol photometer and membrane filter.

limits, namely, silica flour and coal dust when counted with the discriminator set at 0.75  $\mu$ , and cement and sintered iron dust at 1.0  $\mu$ . However, if the discriminator circuit were set at about 1.1  $\mu$  for natural diatomaceous earth and at about 0.5  $\mu$  for flux calcined diatomaceous earth these results would also compare with the impinger counts within  $\pm 15\%$ .

The above discussion was presented to emphasize a point we had suspected from the very beginning of this project—the aerosol photometer must be calibrated against each individual type of dust under study. It is further believed that if the instrument is operated properly, calibrated periodically, and calibrated against various dusts, it will be a more precise method of assessing dust concentrations than the impinger counting technique in present use. It must be emphasized that these statements are based on laboratory tests and apply only to comparison tests with the impinger. Membrane filter counts were higher than the impinger and aerosol photometer counts on three of the dusts tested, namely: coal, cement, and sintered iron. Membrane filter counts were essentially equal to those of the impinger and aerosol photometer on natural and flux calcined diatomaceous earth and silica flour. It is interesting to note, however, that these three dusts are

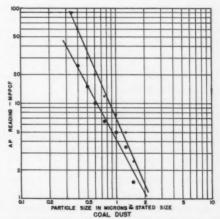


Figure 8. Graph showing numbers of particles at various selected sizes as indicated by the photometer.

the most difficult to count using the membrane filter technique since their refractive index is essentially the same as the oil required to clear the membrane filter.

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high dust concentrations may be encountered at certain operations which would exceed the range of the aerosol photometer even at a 20:1 dilution. Personnel from the Southern Research Institute advised that the counts using the aerosol photometer may be considerably lower than the actual concentration if it is used to sample where the dust concentration exceeds a full-scale deflection with the discriminator circuit set at 0.3 µ. In the case of sintered iron dust, having a median size of 0.4 µ and at dilution ratios to give on-scale readings of 5, 25, and 50 mppcf of 1  $\mu$  and larger size, full-scale deflection was exceeded for 0.3 µ and larger size. However, at these concentrations at 1.0 µ and larger, the AP/MI median ratio was about 1.0 which would indicate, at least with this particular dust, that the AP and MI counts are comparable in spite of the fact that the full-scale deflection was exceeded at 0.3 µ.

#### Particle Size Distribution

Curves prepared from simultaneous membrane filter and aerosol photometer samples of various dusts show that in each case the optical size distribution curve is steeper than the AP curve. The difference between the slope of the AP and optical curve is greater for some dusts than others. These results indicate that the AP is "seeing" particles smaller than are visible under oil immersion. Conversely, it is possible to size larger particles optically and apparently the AP does not "see" these. It must be remembered that only about 10 to 15% of the particles were above 1.5 to 2.0 µ under the test conditions described earlier in this paper. For most dusts tested the median sizes (50% = or < stated size) by both methods were within  $\pm 0.15 \mu$ .

While the size distribution curves prepared from results obtained by the aerosol photometer and membrane filter technique are not parallel, it is believed that for most purposes in the field of industrial hygiene the aerosol photometer will give satisfactory results.

#### General Comments

The aerosol photometer has a number of other features which make it a useful tool in the field of industrial hygiene. As indicated previously, a recorder can be attached to provide an indication of the range and average concentration of aerosol generated during a particular operation. It can be determined from this record and visual observations during an operation, which phases, if any, create more dust than others. Also, a five-pin connector is provided for attaching an X-Y recorder to the aerosol photometer which plots the number of particles equal to or greater than the indicated size.

This instrument gives "on the spot" answers which is important in many instances, especially when one checks the effectivenss or failure of control measures. It is simple to operate and convenient to carry while surveying a plant. A small trickle charger is supplied with the instrument and can recharge the battery overnight. If a source of electric power is available at the sampling site, the trickle charger can be attached and the battery kept at full voltage during use.

A companion instrument is available which, using the output of the aerosol photometer, will automatically indicate the particle size distribution of the aerosol under test.

The aerosol photometer was used for sampling in a mine where wet drilling operations were in progress. A few preliminary tests showed that the aerosol photometer counts were much higher than impinger counts and indicated that the aerosol photometer was counting water mists as well as dust. One method of eliminating this difficulty is to insert a U-tube or other container with a desiceant into the dilution air stream. This dry air evaporates water mists in the sample air.

The aerosol photometer is potentially useful for measuring other aerosols, such as oil mists, pesticides in agricultural spraying, and various smokes and fumes. In such uses the limitations of the instrument must be kept in mind.

#### Summary

A portable light scattering photometer is described and preliminary data are presented to show comparative results of measurements of dust concentrations using this instrument and the impinger sampling and counting technique. Results of preliminary laboratory tests on six types of dust indicate that this aerosol photometer with proper adjustment can be used with the same degree of accuracy as the impinger technique. Furthermore, particle size distribution curves prepared from data using the aerosol photometer and the membrane filter method, while not always parallel, are in good agreement at the median size and check quite closely for about 70% of the particles, i.e., from about 15% to 85%. This instrument has several other features which are useful to industrial hygienists and others.

No extensive field tests have been made using this instrument. It has been useful in studies conducted in a nuclear submarine, in so-called "clean rooms" at a large aircraft plant, in a steel mill, and at a plant involving bagasse dust. Several private companies are currently field-testing an earlier version of this instrument. As time and circumstances permit we will make comparative field tests at various operations.

The present model of this instrument has a few

minor objectionable and troublesome features which can be rectified in future models. The aerosol photometer is not commercially available but one manufacturer is considering production and may have a prototype available within the next two years.

Reference was made earlier that, for purposes of this paper, samples collected on membrane filter were used for comparisons of particle size distributions. Actually, simultaneous membrane filter samples were collected for all the test dusts reported in this paper. Direct duplicate counts were made on a portion of each membrane sample collected and another portion was used for particle size analysis. A number of interesting trends were observed on comparison of membrane filter, impinger, and aerosol photometer counts. It is planned that another paper will be published describing these results. It must be emphasized again that the results reported in this paper are based only on comparisons of the aerosol photometer and impinger.

#### Acknowledgments

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This project would not have been possible without the support and cooperation of many persons. We owe especial thanks to Dr. Lewis J. Cralley, Assistant Chief, Occupational Health Research and Training Facility, for his support and encouragement during the progress of this investigation. Mr. Alfred L. Mendenhall generously assisted in calibration tests, making minor electronic repairs, and focusing the light source of the instrument. Oscar Marsh designed and constructed a protective cover for the instrument's sampling probe. He was usually called upon periodically to clean out the dilution valve assembly. Mr. David A. Fraser assisted in setting up the Wright dust feed apparatus and gave advice on its operation.

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## What is Expected of the Professional Person in Litigation

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RECENTLY I happened to mention to a good friend, who is a physician, that I was going to give a talk on the subject of what a lawyer expects from the professional person in litigation—in other words from an expert witness—and his comment was "I suppose you will tell your audience that you never know what to expect." No doubt he had in mind the old adage "Heaven knows; who can tell? Who shall decide when doctors disagree?"

Speaking seriously I think that a more appropriate title for my remarks would be "What the Lawyer and the Professional Person Expect from Each Other."

I say this because I think the future of our present adversary system of litigating controversies involving medicolegal questions will depend, to an ever increasing degree, on better understanding and more harmonious relationships between the legal profession and the medical and other scientific professions. The relationship between the lawyer and the expert witness involves mutual duties and obligations. The interests of the public and the proper administration of justice make imperative a better understanding of these reciprocal responsibilities.

In speaking today of the "expert witness" I refer not only to physicians, but also to the industrial hygienists, and chemists, the engineerssafety, mechanical, electrical, acoustical, and others—as well as to toxicologists and all others whose special education, training or experience have given them the ability and skill to deal with the increasingly complex problems of our modern industrial life-problems created by such a variety of factors as "fumes, dusts, gasses, mists, solvents, noise, radioactivity, radiant heat and other physical-chemical agents." It will only be from the combined efforts of all of these specialists that we will be able to solve the many problems which lie ahead, such as the prevention of accidents and the elimination of harmful industrial and other types of exposures, the solving of the causes of such accidents and exposures, and

the prompt treatment and rehabilitation of those injured as a result of the manifold hazards of life.

#### Who is an Expert Witness?

Briefly an expert witness is one who "will be deemed qualified if, and only if, he possesses special skill or knowledge respecting the matter involved so superior to that of men in general as to make his formation of a judgment a fact of probative value."2 There is no precise rule governing the manner of acquiring this skill or knowledge-which Wigmore has called "Experiential Capacity"3-and it may result from one or more processes such as the practice of an occupation, observation, special study, professional training, or in fact anything which contributes to the acquisition of accurate, specialized knowledge on a given subject.4 The testimony of the expert generally is received on the theory that it will aid the trier of the facts, whether it be a jury, a judge, or an administrative agency, in reaching the correct conclusions in the issues being litigated.5

#### **Necessity of Cooperation**

The expert, when he assumes the role of a witness, should have a clear understanding of the purpose of his testimony, and of the limitations which the law and the rules of legal procedure impose upon such testimony.

It is the responsibility of the lawyer to fully educate the expert witness upon such subjects. Each profession has a duty of cooperation so that the testimony of the expert will be of greatest value to the trier of the facts.

The increasing recognition of the importance of cooperation between the legal and other professions is evidenced by: (a) the expanded emphasis being placed on the teaching of medicolegal subjects in our American law schools, (b) the new National Interprofessional Code for Physicians and Attorneys recently approved by the American Medical Association and the American Bar Association, and (c) the many symposia and medicolegal conferences held every year which deal with the combined legal, medical

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and scientific aspects of various problems such as ionizing radiation, rehabilitation, etc.

This emphasis on the need of cooperation and solidarity could, and would, be accelerated if the members of the various professions had a better understanding of the basic methods and aims of the respective disciplines and systems involved. While all of these systems seek the discovery of truth and the improvement of the health and well-being of our society, they employ different

methods of approach.7

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The legal profession is dedicated to the principles of equity and justice, and to the application of these principles not only to the judgments and pronouncements, but to the modalities and procedures, of our legal system. It seeks to attain these goals through the medium of advocacy which of necessity involves the presentation of facts, theories, contentions, and arguments.8 On the other hand the physician and the industrial hygienist seek to eliminate occupational hazards and to prevent accident and illness-thereby insuring the physical and emotional health of our population. Their entire approach is scientific rather than polemical, and it is because of the elements of controversy and dissension in the courtroom that many doctors and professional men are reluctant to appear as witnesses. In short they do not completely understand the techniques of law, including the importance of the great legal weapon of cross-examination which is designed to test the credibility and the knowledge of the witness, as well as the probative value of the opinion which he has expressed. I suspect that many medical and professional men prefer to live in a world unhampered by semantics, whereas the lawyer's career is involved primarily with differences of opinion. The honest lawyer seeks to expose fallacious opinion and, where there is honest divergence of opinion, to see that the opinion favorable to his client is convincingly and skillfully presented. Not only are many physicians and other experts today acquiring a better knowledge of legal procedures and courtroom tactics, but there appears to be evidence that the legal profession itself is revising some of its historic concepts as to the proper methods of enforcing legal rights and attaining justice. This is reflected in the following statement which appeared in a recent report of a Joint Committee of the New York State Bar Association, the Association of the Bar of the City of New York and the New York County Lawyers Association:

"... The sporting theory of litigation, with the outcome largely dependent upon skillful use of the twin weapons of surprise and technicality, is certainly no longer in vogue. What generally counts today is not the superior set of wits, but the better case."

Even as the man of science is seeking to open up new frontiers of knowledge for the benefit of humanity so is the lawyer constantly seeking to improve our basic legal system which is intended to draw more clearly the line of demarcation between right and wrong. Difference of opinion is inherent in our whole democratic process, and law is a means of providing the ground rules for the airing and ultimate resolution of these differences. To the pure scientist, probing the unknown, these rules may appear to be forensic shackles but they are indispensable in the regulation of our intricate corporate, social and individual relationships. They must, of course, be revised from time to time to meet the changing needs of our complex society. In spite of the principle of stare decisis, law is by no means immutable and precedent is frequently disregarded, both on the legislative and judicial levels, when it is believed to be in the public interest. Reasonable stability must be sensibly correlated with the need of change and the demands of progress.

#### The Meaning of "Opinion"

Since the basic function of the expert witness is to express an "opinion" on a given set of facts, it would seem appropriate to say a few words as to what we mean by this term. One of my fellow lawyers in Buffalo, recently addressing the Medical Society of the State of New York, quoted the following statement made many years ago by Judge J. M. Longenecker of Chicago: "

"This matter of opinion is a peculiar one, anyhow. First, the expert witness gets on the stand and gives his opinion that if such and such is the case, then such and such must be the result, that is his opinion; then the lawyer gives the jury the benefit of his opinion as to the opinion of the expert; and then the jury are called upon to give their opinions of the opinions of the lawyer and the expert, and it is

only an opinion after all."

In spite of the humorous implications of this observation, it is still true today; indeed the expression of "opinions" is deeply imbedded in our whole medicolegal system. It is well, therefore, to remember that the value of any opinion expressed rests upon such considerations as the intelligence of the witness, his knowledge of the subject acquired by his education, his experience and capacity for observation, his power of reasoning, and above all, his integrity and fairness, his previous reputation for veracity and other similar intangible factors." The expert's opinion is, after all, part of the "evidence" in the case and

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the value of such opinion must, of necessity, be evaluated in the light of the character, as well as the ability, of the individual who testifies. Any expert called upon to express an opinion in a litigated matter should bear in mind always that he is, in effect, an arm of the Court (or of the administrative agency) in the promotion of justice.

#### Importance of Preparation

Preparation for the trial of any case is vitally important.

As a student attending law school I recall an informal talk given by one of the country's outstanding trial lawyers who received a fabulous fee for defending a man of national fame and great wealth who was indicted for certain alleged fraudulent financial activities. His method of preparing this case for trial, as he explained it, is something I will never forget. First he painstakingly interviewed the witnesses and assembled all the facts which were put into a detailed memorandum in their chronological order. He then made an exhaustive study of the law and of the pertinent reported cases, particularly those which were favorable to the contentions of his client. The results of this study were put into a separate, detailed memorandum or brief on the law.

Many lawyers would have stopped at this point believing that they were ready for trial, but not this astute member of the bar. His next step was to put away all the material he had prepared and to pretend theoretically that he was the prosecuting attorney. Proceeding upon this assumption he again fully prepared the case for trial, both from the factual point of view and on the basis of legal research, and the results of this work were again reduced to the form of written memoranda.

Thus, when the case came to trial, he had a thorough knowledge of every possible contingency or argument which might arise. In any event he won the case for his client and, from the way he told the story, I feel sure that his tireless and indefatigable preparation had much to do with the successful result which he obtained.

The amount of preparation of any given case will, of course, vary depending on the importance of the issue to be litigated.

#### Preparation by the Expert

The preparation of the expert witness should include one or more pre-trial conferences with the attorney, and there is no longer any doubt as to either the desirability or the propriety of such conferences. As stated in the National Interprofessional Code for Physicians and Attorneys: 12

"It is the duty of each profession to present fairly and adequately the medical information involved in legal controversies. To that end the practice of discussion in advance of the trial between the physician and the attorney is encouraged and recommended. Such discussion should be had in all instances unless it is mutually agreed that it is unnecessary.

"Conferences should be held at a time and place mutually convenient to the parties. The attorney and the physician should fully disclose and discuss the medical information involved in the controversy."

This statement applies equally to any expert.

#### **Purposes of Pre-trial Conference**

The pre-trial conference between the attorney and the expert witnesses serves a dual purpose, namely:

(1) It will acquaint the witness with the legal considerations, procedures and issues involved. At such a conference the attorney can, and should, explain to the witness the purpose of the cross-examination to which he may be subjected, and which may be designed either to discredit his qualifications, to show that he was biased, or to establish that his opinion was not supported by sufficient data or was contrary to the recognized medical or scientific authorities on the subject.13 He can warn the witness against possible misleading, designing or embarrassing questions which may be put to him. He can tell the witness that if any question propounded to him is not clear, he should so state and ask to have it clarified or rephrased. The attorney can also, if time permits, actually go over his testimony with him and give him an example of the possible crossexamination which he may expect. In general he can help the witness to fully understand the factors involved in the conduct of the litigation.

(2) Simultaneously the expert witness must educate the lawyer in the scientific aspects of the case so that, upon the trial, the latter can elicit the facts and the professional opinion which will best protect the interests of his client. The expert should call the attorney's attention to the pertinent text books and published authorities dealing with the particular problem involved so that the latter will have full knowledge of what has been written on the subject. This mutual effort and understanding, and the interprofessional confidence which it generates, will eliminate much confusion, and at the same time it will make the expert witness realize that the trial is not some sinister and Mephistophelian ordeal to be approached with dread or misgiving.

#### The Hypothetical Question

Since the great majority of medicolegal cases in which expert testimony is necessary involve use of the hypothetical question, an explanation of this legal mechanism might be helpful.

Initially, we must bear in mind that the "underlying assumption of virtually all the cases dealing with expert testimony is that the jury must have the facts upon which the expert bases his opinion in order to evaluate the worth of the opinion..."

If the facts upon which the expert bases his opinion are not within his personal knowledge, or even where he may have personal knowledge, a hypothetical question is generally necessary. The jury or court or trier of the facts must know upon what the expert bases his opinion. It is not necessary that this hypothetical question include all the facts in evidence although it should, generally, include the salient or key facts. The attorney propounding the question may assume the existence of any state of facts which the evidence fairly tends to justify and which is consistent with his theory of the case.<sup>15</sup>

However, it should be borne in mind that any question which assumes facts which have no support in the evidence is improper, and should be excluded or disregarded.<sup>16</sup>

The expert's answer to the hypothetical question put to him must be predicated exclusively upon the facts contained in the question. He may not be asked to express his opinion upon the facts "as shown by the evidence," or based upon "all the evidence in the case." The reason for this is that the trier of the facts (the jury, the court, or the administrative agency) would, under such a broad hypothesis, be unable to determine the precise facts relied upon by the expert, and, of course, this is particularly true in cases where there is a dispute as to the correct facts.

One of the values of the pre-trial conference is that it can help to develop the true case history thereby enabling the attorney to prepare a proper hypothetical question in advance of the trial. And, incidentally, when this is done, it is always helpful to the expert if the question so prepared is given to him before he testifies so that he can study it at his leisure.

I have in some of my own more important cases over the years prepared a hypothetical question documented by specific references to the pertinent scientific and medical data, including entries in hospital records, x-ray findings, laboratory reports, etc. The value of such a question is that in the event of any dispute on the trial as to the correct facts, the source material is readily available, and furthermore a carefully documented question is often psychologically impressive

The length and detail of the hypothetical question will, in each instance, depend on the seriousness and the potential dollar and cents value of the case. Incidentally, I hope none of my questions have been as verbose as the one about which the following story is often told in legal circles. An attorney propounded a hypothetical question to an expert witness which took two days to ask. When he finished the expert said, "I'm sorry, counsellor, but would you mind repeating that question".

Another consideration involved in connection with the use of the hypothetical question should be mentioned. Many hypothetical questions contain flaws, or may omit statements vital to the conclusion to be expressed by the expert witness. If this occurs during the trial of the case, the expert is wholly within his rights in asking that the question be amplified or rephrased to correct any deficiencies. From the point of view of the proper administration of justice reasonable supplementation of inadequate or imperfect hypothetical questions is highly desirable.

#### Qualifications of the Expert

It is important in the trial of every case that the qualifications and prior experience of the expert be fully stated. These should be made a matter of record even though in many cases the opposing attorney may offer to stipulate the qualifications of the witness. The reason for this is that although the witness may be well known to those conducting the trial locally, he may not, if the case involves a jury trial, be known to the members of the jury. Apart from this there is always the possibility of an appeal to a higher Court the members of which will review the record and may not possess any knowledge as to the qualifications of the expert, and which may be important in their evaluation of the case.

The expert should never be asked generally to state his qualifications thereby compelling him to give a long-winded answer as to his education, medical, hospital, and professional affiliations, etc. Any such answer tends to sound egotistical and immediately places the expert in a disadvantageous light.

In my opinion the best method of qualifying an expert witness is to ask him a series of questions dealing with the separate subjects of (1) his profession, (2) his education, including his post-graduate work, (3) the nature of his specialty, (4) his hospital or other affiliations, (5) the medical or scientific societies to which he belongs, etc.

While modesty is an essential attribute of any expert witness, this should never cause him to fail to recite fully his qualifications in his chosen field. As stated by Professor Tracy he "should give the impression of a man who knows his profession but has no desire to brag about his qualifications or unduly exaggerate them."21

#### Conduct of the Expert Witness

There have been many text books and articles written dealing with the subject of the conduct and demeanor of the expert while actually testifying on the witness stand, and the following comments and suggestions are based in part upon some of these,<sup>22</sup> and in part upon my own personal experience and observations.

Since this is a matter involving the human equation and the deep-seated nature of individual personality, since I offer no pretensions of infallibility, and since I thoroughly agree with Emerson that the essence of stupidity is the demand for final opinions, I might add that in the final analysis each expert witness will have to decide for himself the precise manner of his conduct on the stand and how he answers the questions put to him. But here are my observations for what they are worth.

(1) In testifying give some consideration to your personal apeparance and to your general mannerisms on the stand. Dress conservatively. Do not be haughty or arrogant; in short never assume a superior attitude. Be friendly without being smart, and remember that an occasional manifestation of good humor may insulate you against abuse.

(2) Be honest and fair. The proper functioning of our legal system depends on integrity. Evasive answers tend to suppress the truth (suppressio veri).

(3) Express your opinion in a candid way without bias. Any expression of favoritism toward any of the litigants will destroy the value of your testimony.

(4) Never assume the role of an advocate. Leave this to the lawyers, and do not in any way become involved in either the legality or the morality of the claim.

(5) Act with dignity and never lose your temper. An angry witness is always a poor witness. Lawyers can be irritating, especially on cross-examination, but, after all, they are human beings trying to do the best job they can for the clients they represent. As C. Joseph Stetler, Director of the Law Division of the American Medical Association, has pointed out the "typical" trial lawyer is not "an oracle with a silver tongue, who delights in mortifying witnesses and who has a mysterious glamour that winds judges and juries around his finger. Fortunately this 'Perry Mason' type exists almost exclusively in fiction and the movies. Physicians are amazed to find that most

lawyers are quite human, with very normal reactions, such as respect for a brother profession and are apt to regard physicians as their partners in a joint venture—the administration of justice."223

(6) Make your answers brief and to the point. Verbosity can be ruinous.

(7) Never volunteer information which is not responsive to the question propounded. The information you volunteer may be irrelevant and you may be "leading with your chin". The attorneys for the respective parties will generally elicit all the proper and pertinent answers in the questions they ask.

(8) Do not argue with any of the lawyers. Leave the arguments to the attorneys. This is their specialty.

(9) The questions put to you, and the answers you give, should be stated in terms of "reasonable (e.g., medical) certainty" or "reasonable probability". In general, doubtful and speculative expressions of opinion are of no value, and are inadmissible in evidence if properly objected to. However, frequently, and particularly on crossexamination, you may be asked if some conclusion, other than the one you testified to on direct examination, is not "possible". Generally the best answer to such a question is that anything is possible but that you do not believe that such a conclusion is warranted in the case being tried because (again giving the essential basis for your opinion). This not only disposes of the question but it gives you a second opportunity to express your views in the case.

(10) If you are satisfied that your basic opinion, predicated on the facts, is sound, do not give equivocal, ambiguous or dubious answers. Vacillation erodes and destroys the probative value of opinion evidence.

(11) If you do not know the answer to a question, say so frankly. Sincerity will never weaken the value of your testimony and it is always better than offering a hasty or spontaneous "guess."

(12) If a fact, previously unknown to you, is called to your attention, do not be afraid to admit it. As Sir John Collie pointed out you may well be able to demonstrate "that it is not so in reality," and, furthermore, that in any event "the admission will demonstrate such fairness that the remainder of your evidence will have an enhanced value." You can always point out, if it is true, that even assuming the correctness of such fact, it does not change your original opinion because (again giving the basic reasons for your initial conclusion).

(13) Never be nervous or apprehensive. To again quote C. Joseph Stetler "There is no real magic about testifying. Just remember that a

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courtroom is a place where practical men are engaged in the serious work of endeavoring to administer justice. The honest physician who comes to court to tell the truth has nothing to fear."

(14) Insofar as possible, avoid the use of complicated technical and scientific language. This suggestion consistently stressed by all the writers. is a matter I would particularly like to emphasize. Most jurors, and the lay personnel of our administrative agencies, seldom understand the occult and baffling terminology of the highly trained scientist. In most instances language of this type is "over their heads." So, whenever possible, simplify your language. For example, instead of using the word "presbycusis" refer to "loss of hearing due to aging"; instead of "hemiplegia" say "paralysis of one side of the body"; instead of "ecchymosis" say "black and blue mark" or "discoloration of the skin," etc., etc. If you do have to use technical language, explain it. We must do all we can to counteract the drift toward incomprehensibility in the area of human communication suggested by a definition which I recently heard of the word "psychology," namely: psychology is the science which is teaching us a lot of things which we already know in language which we can't understand."

(15) Be thoroughly familiar with the medical or other scientific treatises dealing with the problem about which you are going to testify. The use of learned treatises in the cross-examination of expert witnesses varies considerably in the different states, and in many jurisdictions the Court or examiner has wide discretion as to the extent to which they may be used.26 Apart from this your familiarity with the so-called authorities will enable you, if questioned about them, to show that actually they support your opinion, or in any event that they can be distinguished or are not pertinent to the precise matter at hand. Of course, if you are not familiar with some article or treatise cited, do not hesitate to say so as nobody today can be expected to have read everything dealing with a particular subject; also in many jurisdictions your admission that you are not familiar with the article will bar the crossexaminer from further pursuing this line of questioning. If you are familiar with the article, always be on guard when an isolated sentence is read to you because, removed from its context, it may be misleading; ask to see the complete article. Check the date of publication to see if it is the latest edition; medical and scientific opinion does change with the passage of time. If you disagree with the conclusion of some author whose writing is quoted to you, do not hedge; say so frankly and point out why you disagree with him. If you know of other authorities who

support your position, cite them. And you can always conclude by pointing out that your own opinion reflects your best judgment based not only on what you have read, but on your own personal experience and study of the problem.

If a few of the thoughts which I have suggested are accepted by my good friends in the medical and scientific professions, perhaps the attitude of the expert witness toward a court appearance voiced by Dr. Sidney Shindell of Connecticut could be ameliorated. Dr. Shindell stated:<sup>27</sup>

"To the physician, the courtroom means wasting valuable time to give a carefully restricted opinion, necessarily based on inadequate observation, for persons who cannot understand the details of the problems and who probably will not believe him anyway."

#### Role of the Industrial Expert

Today, many doctors, industrial hygienists and other experts are employees of large corporations, and they are frequently called upon to testify in cases in which their own company is involved. Because of their vulnerable position, based upon the human tendency to view anybody who is on the payroll of a defendant corporation as "prejudiced," witnesses falling in this category have an added responsibility to demonstrate the highest integrity and utmost fairness.28 In the long run. and even though this may result in the loss of an occasional case, the increased respect for the witness flowing from such an attitude should pay handsome dividends. The corporate employee in this category must assiduously avoid any show of favoritism. He should not let sympathy becloud his sound judgment on grounds of altruistic humanitarianism, nor should he be improperly influenced by the interest of his employer in the result of the case; unless he gives his honest opinion, the purpose of our laws would be destroyed.29

In non-controverted cases, particularly in the field of Workmen's Compensation, the industrial physician must work closely with the family doctor to bring about the patient's speedy recovery and to rehabilitate him in order to restore his former earning capacity and his proper place in the economic life of the community. This presents a great challenge which is, I feel, being met with continually increasing success. Here again the concept of cooperation as opposed to conflict and self-interest is all important.

#### Conduct of the Lawyer

Since I said earlier that what I wanted to talk about is "What the Lawyer and the Expert Witness Expect from Each Other." a brief word as

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to the conduct of attorneys in examining the expert witness would seem to be in order. In this connection I can do no better than to quote the following language from the National Interprofessional Code for Physicians and Attorneys:<sup>30</sup>

"It is improper for the attorney to abuse a medical witness or to seek to influence his medical opinion. Established rules of evidence afford ample opportunity to test the qualifications, competence and credibility of a medical witness; and it is always improper and unnecessary for the attorney to embarrass or harass the physician."

This code further states that, "The attorney and the physician should treat one another with dignity and respect in the courtroom." Sound guiding principles such as these should be kept constantly in mind. They would do much to effectuate what Dr. Alexis Carrel referred to as the "synthetic approach" so badly needed in this day of overlapping, interrelated, and interdependent professions, sciences, and disciplines.

#### In Conclusion

While the whole medicolegal field is today undergoing many changes reflected by such manifold considerations as the increasing (and in my opinion dangerous) liberality with which our Workmen's Compensation laws are being interpreted and applied, the growth of both statutory and contractual forms of nonoccupational disability insurance, the liberalization of industrylabor collective bargaining agreements covering medical and hospital care, the advent of group medical practice, and the division of medical responsibility "for the diagnosis, care and treatment of patients with other physicians," nevertheless these very changes are, I believe, bringing about what the American Medical Association Journal has referred to as a "new and sweeping doctor-lawyer rapport" and "the promise of less abuse and deficiency in the field of expert medical testimony."32 Dr. Louis M. Orr, President of the American Medical Association, gave voice to this thought in a recent address, when, in speaking of the professions of law and medicine, he said:

"We are not two clashing opponents, but rather two groups of citizens, bearing different arms, but both battling for our ideals. Let us understand and assist one another in this struggle. Our unity and harmony will inspire those we serve."

While it is undoubtedly true that there may still be some lawyers who do not want "unbiased medical testimony" but rather "medical testimony favorable to their case," nevertheless the

demand for ethical, impartial medical opinion is growing. This is reflected in such proposed remedies as the Uniform Expert Testimony Act, and the New York Expert Testimony Project, which have resulted from dissatisfaction with the old system under which the trial is frequently referred to as a "battle of the experts." The basic concept underlying these proposals is the substitution of "objective medical analysis and appraisal" in place of evidence "colored, and calculated to persuade a court or jury to a partisan point of view." ""

The growing demand for impartial medical (and, of course, other scientific) testimony, referred to more and more frequently in the literature, so cannot be ignored because, with the increasing cost of all kinds of insurance, including automobile and public liability insurance, and the expanding cost of Workmen's Compensation, the public has a definite interest in finding the best, fairest, and most economical solution.

As I pointed out in a paper presented last summer at the annual meeting of the American Bar Association, 30 the demand for impartial opinion is becoming increasingly acute in the field of Workmen's Compensation, particularly in those states which permit "unlimited free choice" of doctors, and where liberality of construction is beginning to throw the burden of the degenerative diseases, as distinguished from disability caused by accidental injury, on to the compensation system.40 The evil inherent in this situation is that a finding of compensability ordinarily guarantees payment of the attending doctor's bill by the employer or the insurance carrier, and this at least creates a "temptation" on the part of such a doctor to report or testify that the disability is related to the patient's occupation. This, in turn, forces the employer or insurance carrier, in many cases, to produce evidence to the contrary thereby resulting in another "battle of the experts" which is both time consuming and costly. This consideration is stimulating a demand for more impartial and equitable methods of verifying true compensable disability. Any system which allows the unrestricted selection, treatment, diagnosis and estimate of disability at the instance of one party, and at the same time imposes the obligation to pay the bill on the other party, can, without proper and adequate safeguards, be dangerous.

One possible solution to this problem is that advocated by Associated Industries of New York State, namely substitution of a plan involving "limited" instead of "unlimited" free choice under which the employer would be given some voice in the designation of the lists or panels of doctors

authorized to treat Workmen's Compensation cases.41 This would preserve the free choice principle but would, in effect, prevent it from being an entirely one-sided and partisan method of se-

In any event, viewing the medicolegal problem broadly, the extent or degree to which we can obtain dispassionate opinion on a voluntary basis through cooperation between the professions will lessen the demand for compulsory or arbitrary methods of seeking neutral evaluations. In some states, for example, the state medical associations or societies have created committees to review medicolegal testimony in cases where the testimony is so contradictory as to suggest that one or more of the medical witnesses has consciously deviated from the truth.42

This is a step in the right direction but I would suggest that the legal, medical and other scientific professions could be more vigorous in policing their own membership in order to eliminate, or at least minimize, any tendencies toward distortion and exaggeration, and in the presentation of either spurious claims or wholly unjustifiable defenses to obviously valid claims.43 This can be done by stressing the basic principles of civic responsibility, service to the public, and moral and intellectual incorruptibility. There is "no higher duty upon the part of the upright lawyer and the straightforward physician than that each should protect the honor and integrity of the profession to which he belongs."44 This applies to all the professions. Interprofessional understanding, trust and cooperation can most certainly help to attain these highly desirable goals, and at the same time counteract the pressures for socialization of the independent professions. Voluntary and conscientious adherence to truth by those in all walks of life will always be the most effective means of preventing the growing power of the state from further destroying our individual rights and liberties.

#### Notes and References

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#### CONTROL OF IONIZING RADIATION

THE ENVIRONMENTAL RADIATION LABORATORY of the Institute of Industrial Medicine, New York University Medical Center in cooperation with the College of Engineering will present a course in Medical and Public Health Control of Ionizing Radiation (Course No. 4815) March 6-17, 1961. This course is concerned with outlining the nature and extent of health hazards from ionizing radiation. It has been organized for physicians, public health officials and others with biological interest and background. After a review of nuclear physics, the subject is systematically developed starting with the action of radiation at the cellular level and leading up to the effect on body organs and the population. Newer concepts of biological mechanisms and pathophysiology will be briefly reviewed where necessary to provide a background for understanding radiobiological effects. Particular emphasis will be placed on providing a realistic basis for the operation of occupational and public health radiation control programs.

Course No. 4813, Radiation Hygiene Measurements, will be presented April 17–May 12, 1961. This is a laboratory course for those persons having some acquaintance with nuclear measurement principles whose work requires their application to specialized radiation protection problems. Experiments will include work with neutron sources and mixed radiation fields. Considerable attention will be given to shielding experiments involving gamma radiation, neutrons, and beta radiation. The principles of scintillation pulse height analysis and nuclear tract microscopy are treated. Among other topics developed are the statistical aspects of nuclear counting and electronic principles used in nuclear detection equipment.

Tuition for Course 4815 is \$100 plus \$10 fee for field trip expenses. Tuition for Course 4813 is \$200. For applications or information write to Office of Associate Dean, New York University, Post-Graduate Medical, 550 First Avenue, New York 16, New York.

# Toxicological Studies on Hydrocarbons-VII. A Gravimetric Method for the Determination of Inorganic and Ethereal Sulfate in Urine

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THE ratio of inorganic to total sulfate in urine has been used for many years as a test of exposure to benzene in industry. The analytical method in use today is essentially the procedure first described by Folin in 1905. It is a time-consuming procedure as evidenced by the following excerpts taken from the directions: \*\*.\*\*

"Slowly, (drop by drop) and without stirring or heating, add 10 ml of the barium chloride solution. Cover the flask and allow to stand at room temperature, without stirring, one to three hours.

"Filter each solution (175 ml) through a fine paper and wash the barium sulfate precipitate with about 250 ml of cold water until free of chlorides.

"Transfer the paper to tared crucibles, put into a cold muffle furnace, turn on the current, and heat until all carbonaceous matter is destroyed. Cool in a desiccator, and weigh. Repeat ignition until constant weight of BaSO<sub>4</sub> is obtained."

A minimum of four hours is required for the analysis described,

The method to be described has been used in our laboratory for more than two years. It was developed because of the need for a rapid, simplified, clinical procedure for the determination of urinary sulfates. The procedure developed requires ordinary laboratory equipment and reagents found in the hospital clinical laboratory rather than the specialized equipment of the analytical laboratory. The techniques are simple and easily learned by a technician rather than requiring the special skills of the analytical chemist. The time-consuming steps, such as dropwise addition of barium chloride, allowing the

barium sulfate precipitate to stand one to three hours, washing the precipitate with large volumes of water after a lengthy gravity filtration, ignition of precipitate in a muffle furnace, etc., are eliminated. The volume of urine used is smaller (10 ml instead of 50 ml), and the time required to hydrolyze the organic sulfate is shortened by autoclaving instead of boiling the acidified urine at atmospheric pressure in an open beaker.

A new rapid quantitative pressure filtration technique is substituted for the suction or gravity filtration currently used. A total volume of 10 ml of liquid is filtered in a few minutes. The precipitation and washing of the precipitate are carried out in a tared centrifuge tube, thus requiring a minimum of quantitative transfer procedures. Since the carbonaceous material formed during the acid hydrolysis is removed by filtration prior to the precipitation of barium sulfate, the precipitate is uncontaminated with organic particulates, and requires a simple, rapid drying in an oven rather than a lengthy ignition to constant weight. In our laboratory 12 urinary sulfate ratios are run routinely in less than four hours by one technician, averaging 20 minutes per sample.

#### Description of Method

#### Chemicals and Reagents

- Approximately 6N HCl. Dilute concentrated reagent grade hydrochloric acid with an equal volume of distilled water.
- Barium Chloride Solution. Dissolve 240 grams of BaCl<sub>2</sub>·2H<sub>2</sub>O in water and dilute to one liter. Filter if necessary before use.
- 3. Acetone. Analytical reagent quality.
- 4. Distilled Water.
- Sodium Bicarbonate Tablets (5 grain (0.325 gm) Eli Lilly & Co., Indianapolis, U. S.)

Presented in part at the Twenty-first Annual Meeting of the American Industrial Hygiene Association, Rochester, New York, April 27, 1960.

#### Equipment

- 1. Volumetric pipettes or 5-10 ml syringe.
- 2. 15-ml conical centrifuge tubes, numbered with glass marking point.
- 3. Centrifuge, clinical model (International), fitted with horizontal swinging head, and shields to accommodate 15-ml tubes.
- Oven, set at drying temperature of 100– 110°C.
- 5. Analytical balance.
- 6. One-ounce, narrow-mouth bottles.
- Burette, Volupettor or automatic dispensing device to deliver 5 ml of hydrochloric acid.
- 8. Cotton-tipped wood applicators.
- Glass stirring rod, 1-2 mm diameter, 6 to 8 inches in length.
- 10. Pressure cooker or autoclave.
- Plastic pressure filter device, designed to fit one-ounce, narrow-mouth bottle, see Figure
   (This device is available from the author.)

#### Procedure

- Add 5.0 ml of urine to each of two oneounce, narrow-mouth bottles. Label one I (Inorganic) and the other T (Total).
- To each bottle add 5 ml of 6N HCl. An automatic dispensing device, such as the Volupettor or Palo Pipetter, is satisfactory for this purpose.
- Autoclave or pressure cook bottle T for two minutes at 15 lbs. pressure. If a pressure

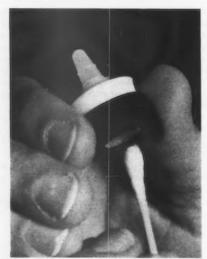


FIGURE 1. Placing cotton-tipped applicator into plastic cone spout.



FIGURE 2. Pressure filtration unit attached to tared centrifuge tube.

- cooker or autoclave is not available the "total sulfate bottle" may be placed in boiling water for 30 minutes.
- While the hydrolysis of ethereal sulfate is in progress in bottle T, proceed with the analysis of inorganic sulfate (I) as follows.
  - (a) Fit a cotton-tipped wood applicator (about two inches in length) into the tip of the plastic pressure filter device as shown in Figure 1. Rotate the applicator while advancing it into the tip to make a tight fit.
  - (b) Drop one-half of a 5 grain sodium bicarbonate tablet into the bottle containing the acidified urine and quickly attach the pressure filter funnel (Figure 2).
  - (c) Invert the bottle with pressure filter attached and insert filter spout into a tared centrifuge tube (Figure 2).
  - (d) When filtration is complete, (Figure 3) add 2 ml of barium chloride solution to the filtered urine in the centrifuge tube, using the graduation marks on the tube to measure the volume of barium chloride added. A "squeeze bottle" containing the barium chloride is convenient for this purpose.
  - (e) Allow the centrifuge tube to stand for five minutes. Centrifuge the tube at top speed for two minutes. Discard the supernatant liquid, wiping the drop ad-

hering to the lip of the tube with absorbent tissue. Add 8–10 ml of distilled water to the tube. Suspend the barium sulfate precipitate in the wash water by rapidly spinning the stirring rod between the thumb and index finger while the tip of the stirring rod is touching the precipitate in the bottom of the tube. Re-centrifuge the tube for two minutes at top speed.

(f) Discard the supernatant wash water, again wiping the final adherent drop of water from the lip of the tube with absorbent tissue. Add 5 ml of acetone to the tube and re-suspend the precipitate in the acetone in the manner used to wash the precipitate with water in step (f). Centrifuge again at top speed for two minutes. Discard acetone wash, wiping the lip free of adherent liquid with absorbent tissue. Dry the tared tube at 100° to 110°C. for ten minutes and weigh. This gives the inorganic sulfate (I). With a single pan Mettler-type analytical balance the weighing can be completed in a few seconds.

5. The total sulfate aliquot (T) is treated in exactly the same manner as the inorganic sulfate described in step 4. After the hydrolysis is completed the bottle is allowed to cool. This may be hastened by placing the bottles in a refrigerator. If desired, the hydrolyzed urine aliquot (T) may be filtered directly into the same tube used for the inorganic sulfate (I), thus eliminating the need for an additional tared tube.

### Results and Discussion

### Precision and Accuracy of the Method

The repeatability of the method for inorganic and total sulfate using the procedure described above is shown in Table I. The procedure limiting the accuracy is the filtration step, since there is some hold-up of liquid on the walls of the bottle and filtering funnel. This volume does not exceed 0.1 ml which is 1% of the total volume filtered. This error can be eliminated by introducing a washing step, in which 2-3 ml of HCl are added to the bottle and pressure filtration repeated collecting the wash water in the centrifuge tube. Since we are concerned with the ratio I/T, and this small error is made for both the inorganic and total sulfate, this additional step is not considered necessary. If the urine aliquot used for the inorganic sulfate determination remains clear

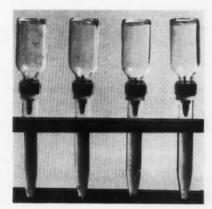


FIGURE 3. Pressure filtration completed. Note black precipitate collected on filter.

Table I
Repeatability of Inorganic and Total Sulfate
Determination in Normal Human Urine
(5.0 ml samples)

Sample No.	mg BaSO <sub>4</sub>		
	Inorganic	Total	
1	24.1	26.7	
2	23.4	26.6	
3	23.8	26.5	
4	23.9	27.2	
5	23.9	26.4	
6	23.9	26.8	
7	22.8	26.6	
8	24.5	26.5	
9	22.6	26.5	
10	22.5	26.6	

Table II Interpretation of Urinary Sulfate Ratio in Terms of Degree of Exposure to Benzene

Ratio $(I \times 100)/T$	Degree of Exposure
above 80	negligible-normal
70 to 80	mild-moderate
70 to 30	dangerous
below 30	extremely hazardous

after addition of hydrochloric acid, the filtration step is unnecessary. If the filtration is eliminated the liquid hold-up errors do not cancel out in the ratio I/T, so that I is larger than T by approximately 1%.

### Calculations

The Urinary Sulfate Ratio is calculated according to the following equation:

$$R = \frac{I \times 100}{T}$$

Where: I is the weight in milligrams of barium sulfate obtained in the unhydrolyzed urine aliquot.

> T is the weight in milligrams of barium sulfate obtained in the hydrolyzed urine aliquot.

> R is the Urinary Sulfate Ratio expressed as per cent.

The theoretical limit of R is 100. Values greater than 100 are due to analytical error.

### Significance of Ratio

The interpretation of the urinary sulfate ratio regarding exposure to benzene is summarized in Table II.

### Summary

1. A rapid simplified gravimetric method has been described for the determination of inorganic and total sulfates in urine.

2. The procedure is suggested as a clinical method for the determination of the urinary sulfate ratio.

3. The analysis can be carried out by a technician in the clinical laboratory since it does not require the specialized equipment of the analytical laboratory or the skills and experience of the analytical chemist.

4. A new rapid quantitative self-regulating pressure filtration technique has been developed for this procedure, which may find wider application in analytical work in which filtration is required.

### Acknowledgment

The author wishes to express his appreciation to Mr. Dennis L. Crow, Products Research Division, Esso Research and Engineering Company, for the photographs.

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### GRADUATE NURSING EDUCATION

THE NATIONAL FUND FOR GRADUATE NURSE Education has been established with a goal of one million dollars for the first year. The fund is intended to give graduate nursing education programs urgently needed support to meet the growing needs for adequately educated nurse leaders. Groundwork for the fund was financed by grants from the Rockefeller Foundation and other sources, following a study by the Institute of Research and Services, Columbia University, of institutional costs of graduate nursing education.

Of 460,000 active nurses in America, about 7,000 hold master's degrees to qualify them for teaching, administrative and other leadership positions. It is estimated that 60,000 nurse leaders are needed. There are 30 fully accredited graduate nursing programs in this country. Presently they confer about 1,100 master's degrees each year, about one-quarter of the number needed. Two schools grant doctorates in nursing.

The first year's goal of the fund will provide only the minimum to give these 30 programs enough additional support to be of real help. The fund has the endorsement of the Secretary of Health, Education, and Welfare, and of the Surgeon General of the Public Health Service. It is also endorsed by the American Medical Association, the American Hospital Association, the American Nurses' Association, the American Council on Education, and the National League for Nursing.

# Tetraethyl Lead Poisoning Incident with Eight Deaths

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THIS paper concerns a tetra-ethyl lead poisoning incident at the U. S. Army Koshiba POL (Petroleum, oils and lubricants) Terminal near Yokohama, Japan. A private company under contract for the cleaning of several petroleum tanks had twenty-three of its employees enter various Japanese hospitals between August 23 and September 10, 1958 following the completion of cleaning Tank No. 6 which contained 115/145 aviation (leaded) gasoline. During the period August 26-September 3, eight of these persons died, including the contractor.

The U.S. Army Petroleum Depot, Japan, had let a contract to a Japanese company for the cleaning of nine underground petroleum storage tanks at the Koshiba POL Terminal. The contract stipulated that the contractor was to assume all liability for his employees, provide for the health and safety of his employees, and equip his men with specifically named items of safety and protective equipment (Figure 1) as recommended by the American Petroleum Institute.1 This contractor had previous tank cleaning experience with various oil companies in Japan prior to accepting the Koshiba contract. Even though he had previous experience, the U.S. Army safety personnel had the safety precautions1 translated into Japanese and had several training classes with the contractor and his workers to impress them with the danger of exposure to tetra-ethyl lead in tank cleaning.

Work commenced on the tanks on February 4, 1958. Six tanks had been cleaned by July. Four of these tanks had contained leaded gasoline and two contained JP-4, a non-leaded jet fuel. The cleaning of Tank No. 6 was started on July 19, 1950.

Tank No. 6 is one of the largest petroleum tanks in the world. It was built by the Japanese for their Navy before World War II. It is entirely underground, with steel walls and a concrete floor. Its diameter is 125 feet and its depth is 95 feet. Its capacity is 203,000 barrels. There are two openings at the top of the tank. One opening used for entry is about four feet in diameter and the other, which is smaller, is used as a vent. The petroleum product is introduced

into the tank and withdrawn from the tank by means of pipes and valves under the floor in a tunnel.

Prior to cleaning Tank No. 6, it was drained and then flushed with sea water. Sea water was again pumped into the tank to the top to force out the vapors and provide support for rafts which were built and placed in the tank to provide platforms for the workers as they cleaned the side walls and the roof structures. One raft in the center served as a landing stage and held an air manifold to distribute air under pressure to the workmen's respirators. Other rafts rode to the sides of the tank to support workmen. Due to the water in the tank, the men wore life vests. They also wore white colored overalls, neoprene boots (hip length), neoprene gloves, and air supplied respirators (Figure 1). After the tank walls were cleaned above the initial water level, the water level was lowered another five or six feet and another wall area was cleaned. This procedure was followed until the walls were thoroughly cleaned. High pressure streams of water were used to knock deposits off other structures inside the tank.

Sludge was removed from the bottom by scraping it, wet, into 55-gallon drums and hoisting the drum in a cage out of the manhole to the surface. The sludge was then transferred to a cement buggy and wheeled to a pit dug for this purpose about 150 feet from the tank opening.

During the tank cleaning, the U.S. Army personnel had warned the contractor that certain employees were failing to wear their respirators at all times when inside the tank. The contractor dismissed some of his employees for these infractions. Others were reprimanded, but permitted to continue working. The contractor himself was warned that he had failed to wear the respirator at times. The contractor's inspector shrugged it off as not important as the contractor was going to be inside the tank only a short time.<sup>2</sup>

Tests were made twice a day for hydrocarbon vapors using a type of combustible gas indicator to determine if vapors were in the explosive



FIGURE 1. Personal protective equipment worn by workers cleaning fuel storage tanks at POL Terminal is shown as well as a cement buggy of the type used for transporting sludge.

range. Too often, personnel engaged in this work are of the opinion that work is dangerous only when the vapor readings are in the explosive range. However, they do not fully appreciate the fact that the tetra-ethyl lead vapor and dust concentrations are not measured by the explosimeter, and also that a potential tetra-ethyl lead hazard by inhalation or skin absorption is always present even though the explosimeter indicates a negative reading for hydrocarbon vapors. In other words, a gas free tank is not necessarily a lead free tank. In fact, the tetra-ethyl lead vapor and/or dust concentration continues to increase in magnitude as the cleaning progresses.

Prior to August 25, 1958 on which date Tank No. 6 was completed, one worker reported to work on August 14, 1958 feeling ill. He was advised to see his physician and the Japanese doctor reported no tetra-ethyl lead involvement. However, on August 26, one worker died and six were admitted to Japanese hospitals within the next few days. A total of twenty-three were under treatment and by September 2 eight had died.

Victims in this Koshiba incident were admitted to various hospitals in Yokohama and the neighboring area. The patients "complained of abdominal cramps, nausea and vomiting, anoxia,

dizziness, hyperhidrosis, pain in the joints, increasing nervousness, anxiety, bad dreams at night, insomnia, and other lesser symptoms. It was observed that progression of this illness eventuated in such objective symptoms as tremor, increased reflexes, mild to moderate hypertension, ataxia, disturbance of speech, disturbance of gait, and in the terminal phases of fatal cases, fever and generalized convulsions."

Medical personnel of the U.S. Army ruled out other possibilities as to cause of illnesses and death, such as botulism, Japanese B Encephalitis. poisons that might be indulged in by such a group in a methyl alcohol drinking party. narcotics, or an accidental group poisoning by chemical cleaning, food containers or insecticides. By comparing available information as to the habits and activities of the personnel during the period preceding the onset of illness, nothing of value was revealed which would incriminate any of the above mentioned possibilities other than tetra-ethyl lead poisoning. The only activity really common to this group was the tank cleaning itself.3 Hospital laboratory findings indicated that the lead content in urine and blood was high.

Observations Made at Storage Tank \*6 on August 30, 1958

The contractor had purchased four electrically operated air compressors. These compressors were located about 60 feet away from the tank's surface hatch opening. At time of observation, it was noted that the compressors were located on the downwind side of the tank. This location of the compressor might permit some contaminatition of air supply source. Two compressors were used at a time, and the other two were for standby purposes. The compressed air flow through a small rubber pressure hose into a filtering apparatus which removed water vapor and oil droplets that might be present in the hose-line air. The air from the two filters (one for each compressor) entered a metal Y-connection to which was attached a larger diameter (about 1 inch) rubber pressure hose. This larger line went down into the storage tank through the manhole port, and then was connected directly to the manifold apparatus mentioned previously. Individual air-line hoses from the manifold were connected directly to each respective respirator face-piece hose connection.

Inspection of the protective equipment after the deaths revealed that the condition of the respirator equipment was, in general, satisfactory. However, some of the boots and gloves had been patched with either hot or cold patches. 60

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the fachad hes. The cleaning of the respirator and other items of protective equipment and clothing was not accomplished by the workers themselves, but was done by a person employed solely for that purpose. Bathing facilities, including hot and cold showers, soap, etc., were provided at the POL Terminal bath house. The cement buggies used for hauling the sludge were heavily covered with sludge deposits.

Subsequent information revealed that the workers in Tank No. 6 thought that this particular tank was cleaner than previous tanks because there were less noxious odors and lower explosimeter tests. Although several of the workers had considerable experience in such tank cleaning projects, they were apparently not fully appreciative of the hazard of tetra-ethyl lead exposures by inhalation or by skin absorption. It was admitted and confirmed later that the workers had removed their respirators at times. How many times or for what duration will never be known.

The records maintained for the workers, showing duration of stay inside the tank and any periods when they were handling sludge, were reviewed to determine if any condition was overlooked that might be an important factor. However, nothing of real value was revealed except that all workers who died had handled the sludge. No conclusion could be made other than the workers did have exposure by inhalation (yapor and dusts) when they removed the air

line respirators while inside the tank, and had skin contact also.

### Conclusions

This tragic accident reported herein did not come about because of the lack of adequate means for protection, but primarily because of the lack of constant and competent supervision which must be provided at all times for tank cleaning work of this nature. Other similar incidents<sup>4, 5, 6</sup> in the military services during the past few years where there have been fatalities, near-fatalities, and serious illnesses from tetraethyl lead exposures make one realize that competent supervision is mandatory for these operations.

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### NEW STANDARD OF LENGTH

THE ELEVENTH GENERAL CONFERENCE on Weights and Measures, meeting in Paris, has adopted a new standard of length based on a wavelength of light, replacing the meter bar which has served as a standard for over seventy years. The new standard became effective on October 14, 1960. Announcement was made from Paris by Dr. Allen V. Astin, Director of the National Bureau of Standards. The Conference also established a central facility at the International Bureau of Weights and Measures for coordination of radiation measurements, and confirmed a new definition of the second of time.

The new definition of the meter is 1,650,763.73 wavelengths of the orange-red line of krypton 86. This will not materially change the measurement of length nor in any way the relation between English and metric units. The inch becomes 41,929.399 wavelengths of the krypton light. This light standard will make it possible for any reasonably well-equipped laboratory to utilize the international basic standard for reference.

The new definition of the second of time was confirmed as 1/31,556,925.9747 of the tropical year 1900 instead of 1/86,400 part of the mean solar days. The possibility of using atomic vibrations as standards for measuring time intervals was discussed. (Let us keep these facts in mind when next we set our watches or buy a ruler.)

# **Experiences with Phase Microscopy**

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#### Introduction

IN ORDER to achieve several objectives which necessitated the determination of the silica concentration of air-borne dusts, the Division of Industrial Hygiene secured a phase microscope and has used it over a period of time. This paper discusses some of the Division's experiences with

this type of microscopy.

According to the Bausch and Lomb Optical Company, "... phase microscopy is a method of illumination in which a portion of the light is treated differently from the rest so as to cause interference with the rest. This is done in such a manner as to cause an otherwise invisible transparent specimen to be visible." Although the phase microscope was originally developed for use in making live biological studies, it has been adapted for industrial hygiene purposes. It has been found useful by the staff of the New York State Department of Labor, Division of Industrial Hygiene in dust counting, particle size analysis, and quantitative analysis of quartzite.

#### Facilities and Procedures

The phase microscope, except for its special condenser and objective, is an ordinary microscope. It is not an especially portable instrument. Its effective operation requires a critically adjusted high powered light source, extremely fine adjustment of an annular ring, careful adjustment of the condenser, and extreme cleanliness.

To perform phase microscopy, the staff used a Bausch and Lomb monocular scope equipped with a phase condenser, a 10× phase objective and a normal 20× eyepiece equipped with a home-made calibrated grid. Illumination was provided by a Bausch and Lomb 6-volt, 18-ampere illuminator. The microscope and illuminator were adjusted in accordance with the Kohler method for illumination and were then clamped to a heavy stand to prevent movement. An annular diaphragm was inserted in front of the condenser. Its image was focused at infinity in the condenser

and was then cast in the rear focal plane of the objective.

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A phase shifting element, i.e., a second annular diaphragm, located in the rear focal plane of the objective caused light which had not been diffracted by the specimen to be reduced in intensity and to be advanced in phase by one-quarter of a wavelength of green light. Light which had been diffracted was gathered by the objective. Part of the diffracted light entered and passed through the center section. This light and that portion which had not been diffracted but had been advanced one-quarter of a wavelength interfered to simulate the phase and intensity distribution which would have been present if the specimen had density rather than refractive index variations.

The next step in the procedure was the utilization of a standard objective to focus the microscope sharply on a dust sample. The removable annular diaphragm was placed in the rack attached to the condenser. Following this, the observer switched to the phase objective and substituted the focusing telescope for the eyepiece.

In use, the annular ring must be accurately centered. To accomplish this, the rack must be moved by means of a screw arrangement while observing the annular ring through the telescope. After the ring has been centered, the telescope is removed and the eyepiece is replaced. Since the ring is not firmly secured, it should be checked periodically to ascertain whether or not it is centered. The condenser must be readjusted for each slide having different thickness since slight differentials will have a significant effect on the resultant image.

### Application

Dust Counting and Particle Size Analysis

Air samples collected in isopropyl alcohol or water were viewed directly in the medium used for collection. The light entering the microscope was filtered to provide a colored tinge. With a green filter, dust particles on a hemocytometer appeared bright to dull white or grey against a light green background. Etched rulings appeared as bright yellow lines. With a blue filter, particles

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appeared black to deep blue against a sky blue background and etched lines appeared white.

A dark green filter is recommended. However, various shades of blue can be used successfully. We found that when using the green filter, eye strain was reduced and we could remain at the microscope for considerably longer periods of

# Samples on Membrane Filters

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For some time, the Division has recognized the advantages to be gained in collecting routine dust samples on membrane filters in lieu of the wet or the impinger methods of collection. Membrane filters can be secured from several manufacturers, in a variety of sizes and weights, and packed in boxes for laboratory loading or in sterile kits. The Division has used Gelman \*1 and \*4 membranes packed in boxes and Millipore membranes packed in both boxes and kits in attempts to collect dust samples for counts. Dust particles can be counted directly on the membrane under the phase microscope provided that an oil having the proper refractive index is used as a wetting agent. Millipore filters become completely transparent when wetted with an oil having a refractive index of approximately 1.507. A mixture of ethyl cinnamate and butyl carbitol adjusted to a refractive index of 1.507 at 25°C was used as the wetting agent to produce transparency. It was noted that there was a tendency for the Millipore filters to soften and dissolve. However, the deterioration did not progress at a rate sufficient to impede completion of the count. Crossman recommends the use of Aroclor 1242 mixed with mineral oil or isoamylsalicylate as a suitable wetting agent for use with Millipore filters.2 This mixture has a refractive index of 1.506.

Attempts to clear Gelman filters have not been successful nor have the efforts to secure low standard blanks with boxed Millipore filters. Apparently the latter are excessively contaminated during their manufacture, storage, or handling. Blanks run on sterile Millipore field monitors have all been relatively low, having from 6 to 12 particles in a field measuring about 350 × 380 microns. For comparative study, samples are being collected in field monitors operated in parallel with midget impingers. Results of the comparative study will be reported at a later date.

### Silica Analysis

The procedure for the analysis of silica utilizes a dispersion staining method. It is necessary to use a mounting medium whose refractive index is equal to that of the material being studied and one in which certain multi-refractive crystals will appear highly colored in comparison to others.

Silicon dioxide, or free silica as it is commonly known, is frequently encountered in nature as opal, quartzite, chalcedony, tridymite, and christobalite. Opal is amorphous; the remainder are crystalline. Quartzite, tridymite and chalcedony are multi-refractive. Christobalite is not. Quartzite has refractive indexes of 1.544 and 1.554; chalcedony 1.533 and 1.539; tridymite has indexes of 1.469, 1.470, and 1.473.

Quartzite appears dark blue with tinges of red when examined under the phase microscope in mounting media having refractive indexes of 1.552 to 1.556. If a polarizing analyzer is used, the blue may sometimes change to red with rotation of the analyzer. Chalcedony and tridymite appear white. Various other crystalline materials, particularly lindesine (a common form of feldspar and beryllonite) and warellite (mixed aluminum phosphate-aluminum hydroxide crystals), have refractive indexes which are very similar to quartzite and produce an almost identical color when stained.

Styrene monomer or a mixture of ethyl cinnamate and butyl carbitol were used as mounting media. The former has a refractive index of 1.554 at 20°C and the latter 1.552 at 25°C. Either produces satisfactory results when placed on thin glass slides; however, both have disadvantages.

Styrene monomer is toxic in low concentrations and is irritating in concentrations as low as 100 ppm. Very small droplets tend to polymerize rather quickly when on slides, and it is essential that performance of the procedure be uninterrupted. Although styrene monomer is procurable in a stabilized form, the time which it may be stored is limited because it will polymerize in a container in about six months. Therefore, it should not be purchased in large quantities. Styrene monomer tends to produce a somewhat better color in the crystals than does the ethyl cinnamate-butyl carbitol mixture. Butyl carbitol is also toxic in equally low concentrations. The mixture of butyl carbitol with ethyl cinnamate is odorous and sickening. It contaminates the person and his clothing and is irritating and nauseating in lower concentrations than styrene, as experienced by Divisional personnel. Although either medium will ultimately produce satisfactory results, the use of styrene monomer is preferable.

It is extremely difficult to clean slides after using either medium. Polymerized styrene droplets are almost impossible to remove; the ethyl cinnamate-butyl carbitol mixture forms a gluelike mass when wetted with water or alcohol. It was found necessary to discard slides and cover glasses after they had been used only once.

Table I Analysis of Dry, Crushed Samples

Sample Numbers	Type of sample	Per cent quartzite as deter- mined by phase mi- croscopy	Silica as determined by chemical analysis	Difference
1-12	Typical rock sam- ples containing silica as quart- zite		Maximum of 3.0	
13	Rock	8.5	3.6	4.9*
14	66	1.4	1.0	.4
15	44	0.3	<1.	<1
16	Talc	1.0	<1.	<1
17	66	10.3	14.6	4.3*
18	46	3.7	4.4	0.7
19	44	9.0	8.0	1.0
20	Gypsum, special neat plaster	2.6	3.0	0.4
21	Gypsum retarder	0.2	0.8	0.6
22	" cement	3.5	0.4	3.1*
23	" stucco	2.8	3.8	1.0
24	" additive	2.1	0.5	1.6

\* Of the twenty-four separate analyses, only these show differences in excess of 3 per cent. None show differences which may be considered as excessive in consideration of concurrent chemical analysis done independently.

Analyses for quartzite are made from samples secured from crushed parent material and from air samples collected in alcohol in impinger flasks or on filter paper. Determinations are made by counting two to three hundred particles, simultaneously differentiating and separating them into two major groups, i.e., quartzite and non-quartzite, and by subsequently classifying them in five separate sections as to particle size within each major group.

Each section of the two major groups comprises a particle size range of respectively 1 to 2, 2 to 4, 4 to 8, 8 to 16, and 16 to 32 microns. Particle size is estimated visually. Discernible color is not visible in particles less than one micron in size and hence these are eliminated. Particles in excess of 32 microns in size are also eliminated because they are atypical. The number of particles in each size group is multiplied by a factor of 1, 5, 10, 20, and 40 respectively. Thereafter, separate totals are obtained for each major group and the percentage of quartzite can be determined by using the following equation:

% quartzite

$$= \frac{\text{total quartzite}}{\text{total quartzite} + \text{total non-quartzite}} \times 100$$

Once sufficient experience is developed in color and size estimation, the procedure becomes routine. If the origin of the sample is known, as was the case with many taken in New York State, it is possible to eliminate from consideration all the alternate forms of silicon dioxide except quartzite. The percentage of quartzite established should equal the percentage of free silica.

The procedure just described was used to make determinations of silica in 24 dry, crushed samples which were concurrently subjected to chemical analysis. Table I lists the various samples and the results of both types of analysis. Microscopic samples were mounted in styrene monomer.

In experimenting with phase microscopy, the Division had as one of its objectives the development of a simple, effective means for determining the silica content of dust samples, particularly those taken during routine air sampling procedures. In order to achieve this objective and to comply with the requirements of both laws and codes, it was necessary first to collect air samples by using standard methods and equipment and to make routine dust counts of each sample prior to making determinations by any other means. This necessitated the addition of several steps to the microscopic procedure itself. For one thing, dust had to be separated from the collecting media and dried. Secondly, a sufficiently large portion of the sample had to be mounted on a clean glass slide.

Crossman's method for separating dusts and mounting samples prescribes evaporation of the medium in a small Pyrex beaker followed by transfer of some of the dust from the bottom of the beaker to a slide. He stresses the importance of collecting a sample sufficiently large to cause the alcohol in which it is collected to appear milky. Since quantitatively adequate samples could not be collected by routine procedures, it was necessary either to adapt Crossman's method or to use an alternate method for separation. Both courses of action were tried.

In adapting Crossman's method, a sample was first transferred from the collecting flask into a dust-free, one-half-ounce vial which served to transport, store and evaporate the sample. The sample was evaporated to a volume of about 0.5 cc in an electrically heated oven set at 95°C. About 2½ to 3 hours were required to evaporate to the volume desired. The remaining volume was poured drop by drop onto a clean slide and the liquid portion was evaporated completely under a heat lamp.

The alternate method employed filtration of the alcohol through a Millipore membrane filter. Using this method, a small chamber was fitted at both ends with filters. The bottom of the chamber, which is large enough to contain about 25 cc, consisted of a filter thimble which was connected

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through a water trap to an air pump. The contents of the impinger flask were introduced into the chamber by removing the top filter holder. After the holder was replaced the pump was turned on and air was allowed to flow through the chamber until the lower-most filter dried. The sample was then examined directly on the filter.

Both procedures have advantages and drawbacks. In using the evaporative method, slides can be read easily and the colors appear true and clear. However, the method is time consuming, tedious, and exposes the sample and slide to dustladen air for a considerable period of time. On the other hand, the filtration method is relatively simple and quick and does not expose the sample to dusty air. However, its use often results in a muddy and unclear appearance of the sample under the microscope. Accurate color determination and particle size estimation by the filtration method is very difficult. Eye strain, fatigue and the likelihood of error is increased. It is felt that the evaporative method is the better of the two.

To date, dust counts and analyses of fifty samples of airborne particulate matter have been made to determine the concentration of quartzite in each. Concurrently, the parent materials from which the airborne matter disintegrated were analyzed chemically for free silica. All airborne samples were collected in isopropyl alcohol using a midget impinger. Preparations for microscopy were made by using either the evapora-

tive or filtration method for drying samples. The results of this work seem to indicate a close correlation between the concentrations of quartzite in the airborne material and the silica in parent materials. However, it is felt that much more work must be done before specific conclusions can be drawn.

### Conclusions

The adaptation of the phase microscope to industrial hygiene techniques is of value in reducing eye strain and increasing clarity of vision while making dust counts and particle size analyses. Longer working periods with increased accuracy are practicable. It is of considerable value in determining with relative speed the quartzite concentrations in parent material and in making similar determinations on air samples. The major difficulties experienced lie in the need for delicate microscope adjustments and in the preparation of microscopic samples from air samples collected in isopropyl alcohol.

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# FATIGUE FROM STANDING

A STUDY TO DETERMINE to what extent the alleviation of the fatigue caused by standing for long periods on hard floors affected the work output, the quality of the work, the number of accidents, the work spoilage, and labor turnover was sponsored by a manufacturer of floor matting. Studies were made of bartenders, supermarket counter girls, and winders in the textile industry. Matting consisting of ½-inch corrugated rubber over a ¼-inch sponge rubber base was used to provide relief of fatigue. The sponge rubber base had a waffle weave pattern molded into top and bottom to create air pockets for greater resilience. Groups of workers provided with the matting were compared with others working on hard flooring.

Among the supermarket counter girls, the number of customers handled per hour during peak hours increased 28% with the use of the matting. Complaints of improper tabulations declined 31%. The girls working on the matting had 40% less wastage, and 53% less absenteeism. They were also more cheerful at the end of the day, and had fewer complaints of backaches.

When the fatigue-reducing mats were provided for spoolers and winders in a textile mill, work output increased 6% but spoilage dropped 41%, and absenteeism was reduced 50%.

Among the bartenders those having the mats were more cheerful and effective, particularly toward the end of the shift. Tavern operators noted a marked decrease in operating costs and an increase in profits believed to be due to the improved attitude and more efficient service of the bartenders when fatigue was reduced by use of the mattings.

# Hazards in the Use of Isopolyesters as Maintenance Coatings

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SOME time ago a test program was initiated at the Richmond Refinery of Standard Oil Company of California, Western Operations, Inc., to investigate the use of unsaturated poly-

esters in maintenance applications.

There were several factors calling for the establishment of the program. The principal factor is the rising cost of maintenance and the need for better materials and methods to combat corrosion. A second motivating factor was the development of unsaturated polyesters based on Oronite Chemical Company's isophthalic acid. These polyesters, commonly known as isopolyesters demonstrated outstanding characteristics. Among these were good adhesion to various substrates and good chemical resistance. By varying the formulation of the isopolyester, one could obtain flexible resins with excellent impact properties; or rigid resins with outstanding high temperature properties and good chemical resistance.

Large scale testing of isopolyesters thus far has been in patching compounds, in construction of laminates, and in surface coatings. The equipment used in laminate construction and an application crew in operation are shown in Figure 1. Note the use of special clothing for personal

hygiene.

Since this large scale test program involves a problem of industrial hygiene, we are presenting here the background information and operating practices followed in these tests to permit safe

usage of isopolyesters.

The improper use of unsaturated polyesters can involve inhalation hazards, skin and eye hazards, and the danger of fire. Our purpose, therefore, is to point out these potential hazards from toxicity and fire and the precautionary measures necessary for safe usage.

#### Resin Make-up

Polyester resins for patching and coating operations usually are received in styrene solutions. Sometimes, additional styrene is added at the job site to further dissolve the resin and to facilitate mixing and application. In setting (polymerization), the styrene combines chemically with the resin to form the final plastic product.

Setting is accelerated by the addition of cure catalysts such as methylethylketone peroxide or benzoyl peroxide. In addition, cobalt naphthenate or dimethylaniline may be added as "promoters" to facilitate setting at room temperature. The mixed resin can be sprayed or applied by brush.

# **Toxicity Hazards**

Inhalation

During polyester resin application, the vapors of styrene can cause concern. The odor may be pronounced and atmospheric concentrations developed during spraying and brushing applications can cause a sweet taste in the worker's mouth and a strong odor of styrene on his breath for several hours after the operation. More severe exposures can cause symptoms of nausea, vomiting, loss of appetite, and general weakness. In laboratory animals exposed to high concentrations of styrene vapor, death has resulted from lung irritation or central nervous system depression. In general, however, the disagreeable odor of styrene at relatively low vapor concentrations, and the eye and nose irritation at higher concentrations, make the inhalation of seriously toxic quantities unlikely unless the victim is trapped in such location that escape from the vapor is impossible. Relating styrene to other common solvents, it is roughly of the same order of toxicity as xylene, and the main concern is with acute poisoning. Styrene apparently does not cause chronic poisoning as does benzene in its cumulative effects upon the blood.

The maximum allowable concentration (MAC) for styrene based on daily eight-hour exposure has been set at 100 parts of styrene vapor per million parts of air (ppm) by volume, primarily on the basis of irritant properties. Atmospheric concentrations of styrene measured during polyester resin operations have been recorded at between 200 and 700 ppm in a large room. These levels undoubtedly would run higher in closed areas, and particularly inside tanks and vessels.

Presented at the Twenty-First Annual Meeting of the American Industrial Hygiene Association, Rochester, New York, April 25-28, 1960.

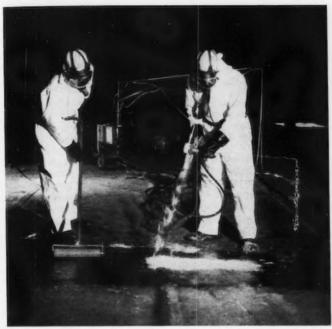


FIGURE 1. Crew applying isopolyester coating by spraying is protected by use of air-line masks, coveralls, neoprene gloves, boots, and by electrical grounding of sprayer head.

During the measurements, the highest concentrations of styrene vapor were recorded during the initial mixing of the resin and catalyst. The next to highest concentrations were recorded during application of the plastic, and from then on the concentrations decreased as the plastic approached complete cure.

If ventilation cannot be applied to the particular polyester resin operation to maintain breathing zone levels of styrene below 200 ppm, personal respiratory protection must be provided. In confined areas known to contain less than 20,000 ppm (2%) of styrene and at least 16% oxygen, a canister gas mask for styrene will suffice. Where the concentration is unknown but estimated to be high, or where the oxygen content may be below 16%, a self-contained breathing apparatus, or an air-line mask, should be used. Respirators are not satisfactory for other than relatively low concentrations because of the irritating effect of higher concentrations of styrene on the eyes.

# Skin Contact

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Styrene also tends to defat the skin, resulting in inflammation and cracking of exposed areas.

The condition often is aggravated by the practice of using acetone to clean plastic from the skin. In order to prevent dermatologic effects, employees exposed routinely to such contact should be provided with gloves and aprons made of neoprene or nonsoluble plastic. Polyethylene gloves have been used with good experience. In operations in which the use of protective gloves would hinder the work, a protective barrier cream (such as Ply No. 9, Fend E, etc.) should be used. The cream should be rubbed well into the skin until dry, covering all exposed surfaces. (It is readily removed with mild soap and water before eating and at the end of the work period.) The eves should always be protected from styrene liquid and vapor by chemical-type safety goggles and/or face shield.

During the initial make-up of the resin prior to application, great care should be taken with those mixtures involving the addition of dimethylaniline promoter. Potentially, this addition could involve an exposure to both the liquid and the vapor. Dimethylaniline acts as a strong depressant on the central nervous system. Since it is readily absorbed through the skin, even small splashes on skin, shoes, or clothing should be

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promptly removed. Peroxide catalysts and cobalt naphthenate also may be injurious upon repeated or prolonged contact with the skin and should be washed off promptly. Protective clothing, including an impervious apron, chemical safety goggles, and an approved respirator are advisable during mixing. The operation should be carried out in a well-ventilated area.

### Fire Hazard

Precautions should be taken to prevent contact of plastic raw materials with combustible organic substances in order to guard against spontaneous combustion. Methylethylketone peroxide is a strong oxidizing agent and, therefore, should be stored carefully to prevent contact with such organic materials. Store in a cool place if possible and carefully note the precautions on the label.

As the plastic polymerizes, it can reach temperatures of 400°F, especially when cobalt naphthenate is added. To prevent spontaneous combustion, wiping cloths should be disposed of in covered metal containers. Such containers can have plastic liners for easy removal and disposal. All excess plastic from squeegee operations should be taken outside as soon as the plastic application is completed. Good housekeeping is imperative to guard against fires, and a final check should be made after every job to determine that all wiping cloth containers and excess plastic constituents are removed from the building or area and disposed of promptly.

Styrene vapor concentrations in the range of 1.6 to 6.1% (by volume) are flammable. Therefore, in confined or nonventilated areas where such concentrations may be formed, sources of ignition from friction, static charge, hot spots, etc., should be guarded against until the resin has cured and the air has cleared. This can be determined by a combustible gas indicator such as the J. W. When the resin is applied by spray gun, the gun head and resin container should be grounded to the metal surface being coated. Similarly, in the initial mixing it is good practice to keep containers in metallic contact when pouring from one to another.

Mixtures of either pure cobalt naphthenate promoter and a peroxide cure catalyst, or of dimethylaniline promoter with a peroxide catalyst, will decompose rapidly and possibly ignite. If premixing in one container is necessary—as may be the case prior to brush application—the cobalt naphthenate or dimethylaniline cure promoter should be thoroughly mixed with the polyester resin before adding the peroxide cure catalyst.

### Summary

The mixing and use of polyester resins can involve exposure to toxic chemicals through inhalation and skin and eye contact. Fire hazards may also be present. Though the hazards will vary with the specific methods of use, the following general suggestions apply:

# Mixing of Resin and Catalysts:

- 1. Conduct mixing in a well-ventilated area.
- Add the "promoters" cobalt naphthenate or dimethylaniline to the polyester resin and thoroughly mix before adding the peroxide cure catalysts.
- 3. Observe special care when using dimethylaniline. To prevent exposure, use an impervious apron, chemical safety goggles, and an approved respirator if the vapors are not controlled. Promptly remove even small splashes of dimethylaniline on skin, shoes, or other clothing.
- 4. Take proper precautions in storing plastic raw materials to prevent spontaneous combustion. Avoid contact of the oxidizing agents and the plastic with any combustible matter.
- For styrene, containers should be kept in metallic contact when pouring from one to another.

### Polyester Resin Application:

- Prevent excessive skin contact. Use a neoprene apron and either gloves or a protective barrier cream on hands.
- Prevent excessive inhalation of the vapors through;
  - (A) effective ventilation of the area.
  - (B) personal respiratory protection:
    - (a) respirators are effective only for relatively low concentrations—not suitable in enclosed areas.
    - (b) in confined areas with sufficient oxygen (over 16%) and less than 20,000 ppm syrrene (2%) a canister gas mask approved for styrene can be used
    - (c) in confined areas with unknown oxygen and estimated high styrene content, a self-contained air-supplied mask or an air-line mask should be used.
- 3. Prevent eye contact. Use chemical-type safety goggles and/or face shield.
- Maintain good housekeeping to guard against fires. Dispose of wiping cloths and excess uncured plastic constituents. Prevent all contact

of plastic materials with combustible materials.

Prevent possible fires. Keep containers in metallic contact when pouring from one to another. Prevent hot spots, friction, and static charges when using resins in confined areas where styrene vapor could form flammable mixtures. When spraying, ground the gun to the resin container and to the metal surface being sprayed.

### STABILIZATION OF FREE RADICALS

THE NATIONAL BUREAU OF STANDARDS has published a new monograph, Stabilization of Free Radicals at Low Temperatures—Summary of NBS Program. This 110-page publication is available at \$1.50 per copy from the Superintendent of

Documents, U.S. Government Printing Office, Washington 25, D. C.

The basis for this volume is a collection of seven papers presented at the Fourth International Symposium on Free Radical Stabilization in Washington, D. C., August 31 through September 2, 1959. The first paper describes the general technical management of the program. This is significant in that the program was based on the belief that maximum productivity would be achieved by a group of competent senior scientists, drawn from government, industry, and educational institutions, provided with adequate research facilities, having the freedom to choose their own research activities, and being relieved of administrative responsibilities.

The remaining papers deal with the technical aspects of trapping, storing, and studying free radicals at extremely low temperatures. Also included are a cross-referenced bibliography of papers published as a result of research completed during the three-year research program, and a summary account of the 1959 Symposium.

Papers included in the publication are: Qualitative comments on the physical and chemical processes in trapped radical systems; Experimental aspects of the NBS Free Radicals Program; A survey of theoretical work on trapped radicals at NBS; Low-temperature chemistry; Methods of production of trapped radicals and properties of radical trapping solids; Identity and concentrations of trapped radicals; and Interactions between trapped species and the matrix.

# A Simple Mathematical Method for Evaluation of Data from Droplet Counts on Settling and Impaction Slides

### ALBERT PFEIFFER

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#### Introduction

DESPITE the development of a variety of instruments for assessing the sizes of droplets suspended in aerosol and spray clouds, a large amount of work is done currently by manually counting and sizing droplet images. Whether the images are produced by the light microscope, by projection of photomicrographs or droplet patterns on transparent slides onto screens, the investigator must adopt a uniform method for the evaluation of the data he gathers. The purpose of this paper is to describe a simple mathematical method which may be used for this purpose.

### Procedure

### Data Obtained Directly

In counting and sizing aerosol droplets deposited on a glass slide, three fundamental numbers for statistical use are obtained. These are (a) apparent particle diameter,  $D_a$ , (b) particle number, N, and (c) area of the slide which is being evaluated, A, on which N particles were counted. These data may be recorded as in Table I.

# Calculated Data

1. The particles are usually combined as they are counted into groups of uniform size limits over the entire range of particles observed. Then the diameter of the droplets in a given group is characterized by averaging in a special fashion the limits of the appropriate group to provide the class diameter, D. From the apparent particle diameter,  $D_a$ , and the spread factor, f, the class diameter, D, is found by substitution in the following equation:

$$D = \sqrt[3]{\frac{D_{al}^3 f^3 + D_{au}^3 f^3}{2}}$$
$$= f \sqrt[3]{\frac{D_{al}^3 + D_{au}^3}{2}}$$

The terms  $D_{\alpha t}$  and  $D_{\alpha u}$  refer respectively to the lower and upper limits of each apparent diameter class. The above formula does two things: it produces a class midpoint which is proportional to the volume of the droplets in the class and it converts apparent diameters of the flattened droplets into diameters of equivalent spherical droplets.

2. From the value of D, the volume,  $V_D$ , of one particle of the specified class diameter is found by reference to a curve or table. Table II gives the  $V_D$  values for particles having a spread factor of DA

3. The *volume* of the drops of a given class diameter per square centimeter,  $C_V$ , and the *number* of droplets of a given class diameter per square centimeter,  $C_N$ , are found as follows:

$$C_V = C_N \times V_D$$
; where  $C_N = \frac{N}{4}$ 

An example of this calculation is contained in Table I.

### Presentation of Data

1. The next step is to construct a curve on rectangular coordinate graph paper, plotting D against the cumulative value of  $C_v$  for each class group. The abscissa (horizontal axis) of this system represents values of the class diameter, D as in Figure 1. From this curve one can easily read the volume median diameter and any relationship between the class diameter D, and the corresponding cumulative values of the volume concentration.

2. A second curve is then constructed on the same coordinate system showing the relationship between D and the cumulative value of  $C_x$  for each class group. From this curve, the value of the number median diameter and any of the other cumulative droplet number values may be read. Now the same basic data are replotted, this time using instead of the cumulative values

of  $C_v$  and  $C_x$  the observed group values against D, in the form of a histogram or bar chart. If the tops of the bars are connected, approximations of the frequency curves for  $C_v$  and  $C_x$  are obtained.

3. The most important data for the frequency distribution of the particles which were measured and counted are now available in one graphical picture, and are shown in Figure 1.

# Statistical Sufficiency

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1. To obtain good statistical values in particle size work one must count particles until an important change of the particle number concentration,  $C_s$ , is not obtained by counting more particles. The statistical principle which requires this procedure is that of sufficiency, and was elucidated by R. A. Fisher's in the provision that every statistical number must be "sufficient", so that no important change occurs by using more data of the same kind. In the evaluation of data from settling and impaction slides, it means that one counts a larger area for large particles and a smaller area for the small ones, so that only the smallest total number of particles necessary is counted.

2. Because the worker must measure exactly the area of the slide or negative which was evaluated, this area should not be too small. This is especially important in assessing slides which contain large numbers of particles per unit area.

3. In general, the area of a slide which is selected for counting should lie along one of its diagonals and extend from one edge to the other, or consist of three or more small rectangles located on a diagonal in order to obtain good average values (Figure 2). For very large drops,

TABLE I Calculation Sheet

D <sub>a</sub> microns	D mi- crons	V <sub>D</sub> microns <sup>8</sup>	N	A cm²	Cy microns <sup>3</sup> / cm <sup>2</sup>	drop- lets/ cm <sup>2</sup>
20-30	10.5	607	90	3	18,210	30
40-50	18.5	3,318	88	2	145,992	44
60-70	26.4	9,641	84	1.5	539,896	56
70-80	30.2	14,433	171	3.0	823,206	57
80-90	34.1	20,778	81	1.5	1,122,012	54
100-110	42.1	39,100	92	2	1,798,600	46
140-150	59.3	109,269	108	3	3,933,684	36
200-210	83.8	308,364	100	5	6,167,280	20
280-300	116.2	822,148	120	10	9,865,776	12
360-380	147.4	1,678,123	120	15	13,424,984	8
440-460	179.9	3,050,877	120	30	12,203,508	4
460-500	192.2	3,720,415	140	70	7,440,830	2

Volume median diameter = 127 microns Number median diameter = 30 microns

Table II
Class Diameter Table
(Class diameters corresponding to apparent diameters)

$\begin{array}{c} \text{Apparent Diameter} \\ D_a \\ \text{microns} \end{array}$		Class Diameter $D$ for spread factor = $0.4^{\circ}$
from	to	microns
0	10	3.2
10	20	6.7
20	30	10.5
30	40	14.5
40	50	18.5
50	60	22.4
60	70	26.4
70	80	30.2
80	90	34.1
90	100	38.1
100	110	43.2
110	120	47.1
120	130	51.1
130	140	55.3
140	150	59.3
150	160	63.4
160	170	67.4
170	180	71.6
180	190	75.6
190	200	79.7
200	210	83.8
210	220	87.8
220	230	91.2
230	240	95.0
240	250	99.3
240a	260	100
260	280	108
280	300	116
300	320	124
320	340	132
340	360	140
360	380	147
380	400	156
400	420	164
420	440	172
440	460	180
460°	500	192

(a) Class limits doubled in size to reduce to a minimum the number of classes required for given particle size range.

(b) To obtain the class diameters for other spread factors, treat the given values as follows: New D = Given D × New spread factor.

it is often necessary to count droplets on the entire slide area to obtain a statistically sufficient drop number. The area which must be evaluated depends on the number of large drops per unit area: the smaller the number per unit area the larger the area which must be examined.

#### Acknowledgment

The author wishes to thank Mr. Robert D. Kracke and Mrs. Helen Lewis for their important help in preparing and proofing the evaluation

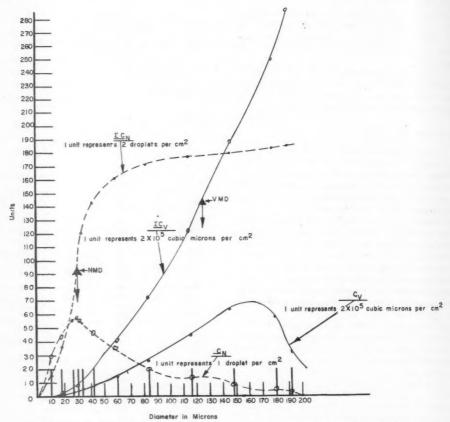
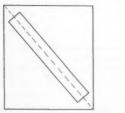


FIGURE 1. Particle number and volume curves and number and volume distribution curves.



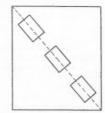


FIGURE 2. Schemes for selecting counting area.

method described and in overcoming the German-English language barrier.

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# HYGIENIC GUIDE SERIES

# Stibine

# (Antimony Hydride)

# I. Hygienic Standards

A. RECOMMENDED MAXIMUM ATMOSPHERIC CONCENTRATION (8 hours): 0.1 part per million parts of air, by volume (ppm).<sup>1</sup>

 Basis for recommendation: Experimental animal studies and by analogy with arsine.

# B. SEVERITY OF HAZARDS:

- 1. Health: Extra hazardous for acute exposures. Exposure of experimental animals has indicated that tolerance to stibine is acquired quite rapidly; however, human data are lacking in this respect. Like arsine, may cause rapid destruction of red blood cells, hemoglobinuria, jaundice, anuria, and death. Subjective effects are manifest as headache, nausea, and weakness. Early subjective signs are headache and nausea, lumbar and epigastric pain, followed by passage of dark red urine a few hours after exposure.
- Fire: Practically none.
   Short exposure tolerance: Not known.
   Exposure of dogs and cats for one hour

to 40-45 ppm has proven dangerous.2

#### **II. Significant Properties**

A colorless gas. Odor described as "characteristic".

Chemical formula: SbH<sub>3</sub>
Molecular weight: 124.78
Boiling point: -17°C

Relative vapor den-

sity: 4.1 (air = 1)

Solubility: 20 volumes in 100 volumes of water (room tempera-

ture)

At 25°C and 760 mm

Hg: 1 ppm: 5.1 mg/m³ 1 mg/liter: 196 ppm

# III. Industrial Hygiene Practice

- A. Recognition: Stibine has been shown to be liberated during the charging of storage batteries, due to the action of nascent hydrogen on the antimony present in the plates.3 The gas may also be liberated when certain drosses are treated with water or acid.4 Antimony-containing alloys may liberate stibine on contact with acid. Although arsine is usually suspected in cases of acute hemolytic phenomena in metal workers, cases so reported may conceivably be partly due to stibine because of the common presence of antimony. Because of the concurrent exposure to both gases in some reported cases, the clinical picture in humans exposed to pure stibine is not clear.
- B. EVALUATION OF EXPOSURE:
  - 1. Instrumentation: No direct reading instruments available.
  - 2. Chemical methods: A rapid semi-quantitative evaluation can be made by means of silver nitrate test papers. A more precise method involves absorption of the gas in mercuric chloride solution, followed by colorimetric determination with rhodamine B.<sup>5</sup>
- C. RECOMMENDED CONTROL PROCEDURES: Keep atmospheric concentrations below 0.1 ppm by enclosure or ventilation.

### IV. Specific Procedures

- A. First aid: Put at bed rest and obtain medical care as soon as there is any knowledge of illness or exposure. Any worker known to have been exposed to high concentrations should be hospitalized at once. There are no specific first-aid measures other than immediate removal from exposure.
- B. Special medical procedures: Obtain blood type and donors. Measure fluid

intake and output of urine. Treat as in other types of acute hemolytic anemia. Give replacement transfusions as necessary. Based on analogy to arsine and the experience with cases of combined arsine and stibine exposure, BAL is considered ineffective and the prognosis after severe exposure is poor.<sup>6</sup>

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# Phosgene

# (Carbonyl Chloride)

# I. Hygienic Standards

- A. Recommended maximum atmospheric concentration (8 hours): one part of gas per million parts of air, by volume (ppm).<sup>1</sup>
  - Basis for recommendation: Irritation of lower respiratory tract in man and animal.
- B. SEVERITY OF HAZARDS:
  - Health: Severe for both acute and chronic exposures. Phosgene affects primarily the alveoli and smaller bronchioles of the lung, rather than the larger bronchi or trachea. Overexposure may result in delayed pulmonary edema, 6 to 72 hours after exposure, or in disability from emphysema, bronchiectasis or bronchitis.<sup>2,3,4,5</sup> Deaths have occurred from military and occupational exposures.
- C. SHORT EXPOSURE TOLERANCE: In humans, exposure to five ppm may cause coughing within a few minutes. Exposure to 10 ppm or more for one minute may result in serious irritation of the lower respiratory tract. One-half hour exposures to concentrations of 41–50 ppm produces fractional mortality in dogs.<sup>2</sup>
- D. Atmospheric concentration immediately hazardous to life: Above 50 ppm may be fatal.

#### II. Significant Properties

A colorless gas with a sharp pungent odor. Chemical formula: COCl<sub>2</sub>

The Committee wishes to acknowledge the assistance of Dr. E. C. Riley in the preparation of this Hygienic Guide.

- Molecular weight: 98.92
- Specific gravity: (of 1.392 at 19/4°C
- liquid) Relative vapor den-
- sity: 3.4 (air = 1)
- Boiling point: 8.3°C Solubility: decomposes in water
  - or alcohol; soluble in many organic solvents

# At 25°C and 760 mm

- Hg:
- 1 ppm: 4.05 mg/m<sup>3</sup> 1 mg/liter: 247 ppm

# III. Industrial Hygiene Practice

- A. Recognition: Used in organic syntheses. The irritant qualities are insufficient to give warning of a hazardous concentration. A trained observer can recognize the odor of 0.5 ppm6 by volume. Various observers have described the odor as that of musty hav or new-mown hay;4 at hazardous concentrations it is not uncomfortable. It is said that a person who has been exposed to phosgene has a definitely unpleasant reaction to tobacco smoke following exposure.4 Phosgene may be formed by the action of ultraviolet radiation (as from inert gas metal are welding)7 on chlorinated solvent vapors in the air and when the decomposition of chlorinated solvent vapor is catalyzed by hot metal or accomplished by slow combus-
- B. EVALUATION OF EXPOSURES: Determination of phosgene in concentrations below one ppm in air is somewhat difficult. Test

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papers and test crayons have been developed to indicate the presence of phosgene in low concentrations.<sup>8, 9, 10</sup> Quantitative chemical methods using diphenyl urea are not reliable in low concentrations. However, a method utilizing ultraviolet absorption in aqueous solution has been developed and seems to permit quantitative air analysis in the one ppm range.<sup>11</sup> The basis for the method is the formation of 1,3-diphenylurea when phosgene reacts with aniline.

C. RECOMMENDED CONTROL PROCEDURES:
Concentrations should be kept below one
ppm. Wherever phosgene is used in organic chemical syntheses or is likely to be
generated in an industrial operation,
personnel should be trained to recognize
the odor and instructed in the use of
protective measures and first aid. Control
is generally provided by enclosure supplemented by local or general exhaust ventilation to remove the gas before it reaches
the breathing zone of the operators. Inert
gas shielded are welding should not be
carried on in areas where chlorinated
solvent vapors may be present.

# IV. Specific Procedures

- A. First aid: Since the immediate reactions to serious exposures are negligible, it is important that all exposed personnel be removed from further exposure and placed at bed rest under medical observation. Anyone who has been exposed to phosgene should be under observation for at least 24 hours to be sure that delayed reactions or symptoms do not occur.
- B. SPECIFIC MEDICAL PROCEDURES:
  - 1. All personnel who may be exposed to phosgene should have complete medical examinations, including chest x-rays, prior to starting work. In preplacement examinations, consider the advisability of excluding from possible exposure those individuals with chronic pulmonary disease.
  - 2. Treatment of acute exposure: An individual who has been exposed to phosgene should be placed at complete bed rest. The patient must then be followed closely for evidence of pulmonary edema or congestive heart failure. In addition to clinical observation, records of the blood pressure, pulse and respiratory rates, total white and differential blood count and platelet

- count may be useful in predicting the extent of pulmonary damage. The use of 100% oxygen may give relief of symptoms. Caution should be observed in administering oxygen under pressure, particularly in cases with impending cardiac failure. 12, 13
- 3. Periodic medical examinations: Individuals who are repeatedly exposed to low concentrations should have routine physical examinations, including chest x-rays and where practical, pulmonary function tests, such as vital capacity and one-second timed vital capacity. This is particularly important in the medical management of those who have experienced acute exposures.

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# n-Butylamine

# (1-Aminobutane)

# I. Hygienic Standards

- A. Recommended maximum atmospheric concentration (8 hours): Five parts of vapor per million parts of air by volume (ppm).<sup>1</sup>
  - Basis for recommendation: Industrial experience under known exposure levels.<sup>2</sup>

# B. SEVERITY OF HAZARDS:

- Health: High acute hazard; moderate chronic hazard. Liquid produces severe damage to skin or eyes. Vapor is irritating to upper respiratory tract and eyes. Inhalation of concentrations at or above the threshold for irritation may produce mild headaches and flushing of the skin of the face. Cumulative effects have not been observed.<sup>2</sup>
- 2. Fire: Flash point 45°F (open cup). Explosive limits 1.7%-9.8%.
- C. Short exposure tolerance: Ten to 15 ppm is highly irritating for short exposures.<sup>2</sup>
- D. Atmospheric concentrations immediately hazardous to life: Unknown for human. 3,100 ppm killed 3 out of 3 rats in 50 minutes, 4 whereas rats survived a single four-hour exposure to 2,000 ppm.

### **II. Significant Properties**

A flammable colorless liquid with a pungent ammoniacal odor.

Chemical formula: CH<sub>3</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>NH<sub>2</sub>

Molecular weight: 73.14 Specific gravity: 0.7401 at 20°C

Boiling point: 0.7401 a

Relative vapor den-

sity: 2.5 (air = 1)

Vapor pressure: 105 mm Hg at 32.2°C miscible with water, alcohol, and ether

At 25°C and 760 mm

Hg: 1 ppm:

 $3.0~\mathrm{mg/m^3}$ 

1 mg/liter: 334 ppm

#### III. Industrial Hygiene Practice

A. Recognition: By its characteristic ammonia-like odor which is easily detectable

The Committee wishes to acknowledge the assistance of Mr. F. A. Miller and Mr. R. F. Scherberger in the preparation of this Hygienic Guide.

by most individuals at 1–2 ppm and is moderately strong at 2–5 ppm.<sup>2</sup> Used in pharmaceuticals, dyestuffs, rubber, chemicals, emulsifying agents, desizing agents for textiles, and photography.

### B. EVALUATION OF EXPOSURES:

- 1. Direct instrumentation: None known.
- 2. Chemical: Collect in acid-isopropanol. Color is developed in an aliquot using pyridine and ninhydrin reagent. The optical density is read at 575 mμ in a suitable colorimeter.³ Also, it may be determined by a procedure similar to the Kjeldahl method⁵ or by using a colorimetric procedure employing anhydrobisindandione.6
- C. Recommended control procedures:

  Maintain air concentrations below 5 ppm
  by adequate ventilation. If unpleasant or
  irritating concentrations are encountered
  in short operations, these can be controlled by the use of personal respiratory
  protective equipment. Eye and skin
  protection should be worn wherever concentrated solutions are handled. Provide
  facilities for flushing skin and eyes.

### IV. Specific Procedures

- A. First aid: Immediately flush skin or eye with large amounts of water. Continue flushing the eyes for at least 15 minutes. Remove all contaminated clothing and flush underlying areas with water. In case of eye contact or inhalation of high concentrations of vapor, the patient should be seen promptly by a physician.
- B. Specific Medical procedures: In preplacement examination, consider possible aggravation of chronic pulmonary or skin conditions.

### V. References

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# Dioxane

# (Diethylene-1,4-dioxide)

# I. Hygienic Standards

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- A. RECOMMENDED MAXIMUM ATMOSPHERIC CONCENTRATION (8 hours): 100 parts of vapor per million parts of air, by volume (ppm).<sup>1</sup>
  - Basis for recommendation: Toxicological observations on animals.<sup>2, 3, 4</sup>
- B. SEVERITY OF HAZARDS:
  - 1. Health: High for acute and repeated exposures. The vapor has poor warning properties and can be inhaled in amounts which may cause serious intoxication or death with injury of the liver and kidneys. 5.9 Several days' exposure to vapor concentrations averaging 470 ppm was judged responsible for a death with central nervous system damage, bronchopneumonia, and severe liver and kidney injury. 5 The liquid may be absorbed through the skin in sufficient quantities to produce injury. 2
  - 2. Fire and explosion: High. Flash point 54°F (closed cup). Explosive limits by volume in air, 2% to 22%; can form explosive mixture on distilling to dryness. When anhydrous, tends to form explosive peroxides in contact with air.
- C. Short exposure to Joderance: Fifteen minute exposures to 300 ppm caused mild transient irritation of the eyes, nose and throat. Animal studies suggest that a single exposure to less than 1,000 ppm for a period not over one-half hour would be relatively safe. A. One-and-one-half hour exposures to 1,000 ppm repeated twice daily for five to sixty-seven weeks produced severe liver and kidney injury, but no deaths in several animal species.
- D. Atmospheric concentrations immediately hazardous to life: Not known for humans. A single three-hour exposure to 30,000 ppm (near saturation) is fatal to animals.<sup>8</sup>

# II. Significant Properties

Chemical formula:	CH2CH2OCH2CH2O
-------------------	----------------

Molecular weight:	88.10
Specific gravity:	1.0356 at 20°/20°C
Boiling point:	101.3°C at 760 mm

	Hg
Freezing point:	11.8°C
Vapor density:	3 (air = 1)

tupor delibity.	0 (an - 1)
Per cent in saturated	
air.	4 75 at 25°C

air:	4.75 at 25°C
Vapor pressure:	37 mm Hg at 25°(
	(approx.)

# At 25°C and 760 mm

Hg:	
1 ppm:	$3.6  \mathrm{mg/m}$
1 mg/liter.	278 nnm

# III. Industrial Hygiene Practice

- A. Recognition: Used chiefly as a solvent for lacquers, paints, varnishes, plastics, resins, dyes and oils. The faint odor and mild irritant properties of the vapor do not give adequate warning of unsafe concentrations.<sup>2</sup>
- B. EVALUATION OF EXPOSURES
  - Instrumentation: Can be determined by use of the interferometer. Mass spectrometry is applicable and gas chromatography offers possibilities.
  - 2. Chemical methods: No satisfactory method for low concentrations.
- C. Recommended control procedures:

  Maintain air concentrations below 100
  ppm by appropriate process controls and
  general or local ventilation. Higher, shortduration concentrations should be
  avoided. The lack of warning properties
  make it necessary to check all areas by
  air analysis. Repeated skin contact should
  be prevented.

The Committee wishes to acknowledge the assistance of Dr. W. L. Sutton in the preparation of this Hygienic Guide.

# IV. Specific Procedures

- A. First aid: In case of a splash on skin and clothing, any contaminated clothing should be removed and the skin washed with water. In case of eye contact, the eye should be flushed with water for 15 minutes. Medical care should be obtained for anyone exposed to significantly elevated vapor concentrations or for anyone exhibiting symptoms.
- B. Special medical procedures: Careful pre-employment and periodic medical examinations with special emphasis on liver and kidney functions are indicated for persons exposed to dioxane. Where there is a hazard of major acute exposures or substantial repeated exposure to concentrations approaching or exceeding the threshold limit value, persons with central nervous system, liver, or kidney disorders should not be employed. Cases of over-exposure should be carefully observed for evidence of liver or kidney failure and early supportive therapy instituted.

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# COMPARISON OF TWO TECHNIQUES FOR MEASURING MICROWAVE ATTENUATION

TWO METHODS OF MEASURING MICROWAVE attenuation have recently been compared at the National Bureau of Standards Radio Standards Laboratory at Boulder, Colo., by a technique that virtually eliminates environmental influences. Both techniques were developed by the Bureau's microwave circuit standards laboratory; one is an ultraprecise power attenuation measurement system, and was used as a reference in testing the second technique, a recently developed subcarrier system for measuring attenuation. These systems are significantly more precise than other methods now in use, and the comparison shows that measurements made with the two agree within 10 microbels.

Both attenuation measurement methods use a two-channel wave-guide system. The low power required by the subcarrier technique may be "borrowed" from a power measurement system by directional couplers without disturbing the latter. Thus the test attenuator can be placed in one channel which is common to both systems. This technique eliminates environmental effects and mismatch errors, and minimizes re-settability limits of the attenuator when making comparisons.

The attenuator used in the comparison was a rotary vane, WR-90 waveguide variable attenuator and the measurements were performed at 9.4 Gc/s. The precision of the modulated subcarrier method was approximately 0.0005 db at low values of relative attenuation and decreased to about 0.005 db at 20 db relative attenuation.

The standard attenuator used in the subcarrier method is a voltage ratio transformer. It is not possible at present to obtain calibrations that are as accurate as is theoretically possible with this type of standard, but the results of this test indicate that the accuracy of the standard is at least comparable to the precision attained by the subcarrier method. Therefore, this particular transformer as well as others similar to it can be used as a standard attenuator in the subcarrier system.

# **Author Index**

Abee, H. H.: Contamination Resulting from the Release of Radioactive Liquid Wastes to the Tennessee River System. April, p. 156

Alercio, John S.: Urinary Uranium Levels in Non-Exposed Individuals. February, p. 68

Allen, C. H.: Critique of Our Present Noise Control Engineering Efforts and Accomplishments. April, p. 130

Allen, Daniel R.: A Mass Loading and Radioactivity Analyzer for Atmospheric Particulates. August, p. 300

Alpaugh, E. L.: Health Aspects of the Commercial Melting of Uranium-Contaminated Ferrous Metal Scrap. April, p. 178 Anderson, Darrell E.: Efficiencies of Filter Papers for Collecting Radon Daughters. October, p. 428

Ayer, Howard E.: Evaluation of an Aerosol Photometer for Dust Counting and Sizing. December, p. 491

Baier, Edward J.: Photofluorographic Study. October, p. 436 Beasley, C. Harold: An Investigation of Lens Opacity on Personnel Operating a Portable Nuclear Reactor. February, p. 84

Belding, Harwood S.: Laboratory Simulation of a Hot Industrial Job to Find Effective Heat Stress and Resulting Physiologic Strain. February, p. 25

Black, R. H.: Protecting and Cleaning Hands Contaminated by
 Synthetic Fallout under Field Conditions. April, p. 162
 Blanchard, Richard L.: Evaluation of Radiation Hazards Cre-

ated by Thoron and Thoron Daughters. June, p. 201
Bless, S.: Retention and Fate of Iridium-192 in Rats Following

Inhalation October, p. 414

Blocker, Hyman: Mycological Contamination of Impinger Dust

Samples. June, p. 261
Bloomfield, Bernard D.: Air Pollution Inventory Procedure and

Results. April, p. 136
Rotsford. James H.: What Do You Mean by Normal Hearing?

Botsford, James H.: What Do You Mean by Normal Hearing? April, p. 125

Brewer, Lial W.: Separation of Fluoride by Ion Exchange: Application to Urine Analysis. August, p. 287

Brief, Richard S.: Multi-Operational Chamber for Calibration Purposes. June, p. 239

 Brieger, H.: Retention and Effect of Radioactive Particulates in the Lung. June, p. 195
 Brugsch, Heinrich G.: Treatment of Chronic Mercurialism with

N-Acetyl-Penicillamine. October, p. 419
Bryan, Fred A.; A Generator for Producing Low Concentrations

of Vapor in Inhalation Chambers. October, p. 423 Butler, H. L.: Protective Clothing Program at the Savannah

River Plant. February, p. 55 Carson, Theophilus R.: The Effects of Continuous Exposure of

Animals to Ethanolamine Vapor. October, p. 374 Casarett, L. J.: Retention and Fate of Iridium-192 in Rats Fol-

lowing Inhalation. October, p. 414

Charsha, R. C.: Development of a Freeze-out Technique and

Constant Sampling Rate for the Portable Uni-Jet Air Sampling

Constant Sampling Rate for the Portable Uni-Jet Air Sampler. August, p. 325

Cholak, J.: Determination of Manganese in Air and Biological

Material. October, p. 356

The Concentrations of Oxidant (Ozone) and Nitrogen Dioxide

in the Air of Cincinnati, Ohio. December, p. 452 Christie, H.: Acute Inhalation Toxicity of Vinyl Chloride to Laboratory Animals. October, p. 394

Christman, Robert P.: Survey of Current Status of Raynaud's Phenomenon of Occupational Origin. February, p. 80

Church, Franklin W.: Multi-Operational Chamber for Calibration Purposes. June, p. 239
 A Criterion for Evaluation of Noise Exposure. December, p.

481 Clay, Maurice F.: Treatment of Exhaust Gases from Boron Fuels. April. p. 110

Clayton, George D.: A Study of the Relationship of Street Level

Carbon Monoxide Concentrations to Traffic Accidents. February, p. 46

Clayton, J. Wesley, Jr.: Toxicity Studies with Octafluorocyclobutane. October, p. 382

Cogswell, Richard V.: Use of Spider Silk in Aerosol Research. June, p. 263

Cook, Warren A.: A Study of the Relationship of Street Level Carbon Monoxide Concentrations to Traffic Accidents. February, p. 46

Cottrell, W. D.: Contamination Resulting from the Release of Radioactive Liquid Wastes to the Tennessee River System. April, p. 156

Cowing, Russell F.; Radiation Dosage to Medical Personnel. April, p. 169

Danziger, H.: Acute Inhalation Toxicity of Vinyl Chloride to Laboratory Animals. October, p. 394

Delaplane, M. A.: Toxicity Studies with Octafluorocyclobutane. October, p. 382

Donati, Edward: The Sub-acute and Chronic Toxicity of 1,1-Dimethylhydrazine Vapor. June, p. 207

Downing, Theodore O.: The Effects of Continuous Exposure of Animals to Ethanolamine Vapor. October, p. 374

Downs, William L.: Acute and Sub-acute Toxicity Studies of Thallium Compounds. October, p. 399 Dubois, L.: Survey of Lead Hazards in Indoor Firing Ranges.

June, p. 256

Duffee, R. A.: A Tracer Technique to Measure Deposition of

Stack Emissions. October, p. 343
Elbert, Walter C.: Benzo(a) pyrene Content of the Air of Ameri-

can Communities. December, p. 443
Elkins, Hervey B.: Treatment of Chronic Mercurialism with

N-Acetyl-Penicillamine. October, p. 419 Enders, John W.: Radioactive Trash Disposal at Los Alamos.

April, p. 151

Fassett, David W,: The Determination of n-Butylamine in Air.

Passett, David W.: The Determination of n-Butylamine in Air. December, p. 471 First, Melvin W.: Air Flow Resistance of Flexible Metal Hose.

First, Melvin W.: Air Flow Resistance of Flexible Metal Hose.
August, p. 296

Fisher, A. M.: Acute Inhalation Toxicity of Vinyl Chloride to Laboratory Animals. October, p. 394

Fosdick, Lee B.: Hazards from the Use of Isopolyesters as Maintenance Coatings. December, p. 522

Fox, Francis T.: Benzo(a)pyrene Content of the Air of American Communities. December, p. 443

Fredrick, W. G.: A Study of the Relationship of Street Level Carbon Monoxide Concentrations to Traffic Accident. February, p. 46

Fugman, Frank S.: Constant Flow Regulators for the High-Volume Air Sampler. April, p. 115

Gerarde, Horace W.: Toxicological Studies on Hydrocarbons— VII. A Gravimetric Method for the Determination of Inorganic and Ethereal Sulfate in Urine. December, p. 511

Gilbert, Harry: Determination of Mercury in Blood. December, p. 475

Gill, W. E.: Determination of NO<sub>2</sub> and NO in Air. February, p. 87 Goddard, J. W.: Retention and Effect of Radioactive Particulates

in the Lung. June, p. 195 Goldwater, Leonard J.: Determination of Mercury in Blood.

December, p. 475
Goren, Sidney: The Inhalation Hazard from Dust-producing

Goren, Sidney: The Inhalation Hazard from Dust-producing Operations in the Repair of Ships with Low-level Radioactive Contamination. February, p. 71

Graul, Robert J.: A Spectrophotometric Method for the Determination of Mercaptans in Air. Dec., p. 466

Greene, Earle A.: The Sub-acute and Chronic Toxicity of 1,1-Dimethylhydrazine Vapor. June, p. 207

1

- Groff, William A.: The Effects of Continuous Exposure of Animals to Ethanolamine Vapor. October, p. 374
- Hammond, J. W.: Industrial Hygiene Features of a Petro-Chemical Benzene Plant Design and Operation. April, p. 173
- Harrison, Walter K., Jr.: Constant Flow Regulators for the High-Volume Air Sampler. April, p. 115
- Harrold, Gordon C.: The Expert Witness. August, p. 330
  Harwood, John F.: An Application of Gas Chromatography to
- Analysis of Solvent Vapors in Industrial Air. February, p. 20 Hauser, Thomas R.: Benzo(a)pyrene Content of the Air of American Communities. December, p. 443
- Held, B. J.: Health Aspects of the Commercial Melting of Uranium-Contaminated Ferrous Metal Scrap. April, p. 178
   Helwig, Harold L.: A Spectrophotometric Method for the Deter-
- mination of Mercaptan in Air. Dec., p. 466
  Hermann, E. R.: Industrial Hygiene Features of a Petro-Chemi-
- cal Benzene Plant Design and Operation. April, p. 173 Herrick, Robert A.: Concepts in Fabric Air Filtration. February,
- Herrick, Robert A.: Concepts in Fabric Air Filtration. February, p. 1 Hertig, Bruce, A.: Laboratory Simulation of a Hot Industrial
- Job to Find Effective Heat Stress and Resulting Physiologic Strain. February, p. 25
- Hill, William H.: Determination of Pentaborane in Air by Means of Activated Carbon. February, p. 15
  - Determination of Decaborane and Pentaborane by Means of Triphenyltetrazolium Chloride. June, p. 231
- Determination of Decaborane by Means of Ferricyanide. August, p. 312.
- Holaday, Duncan, A.: Evaluation of Radiation Hazards Created by Thoron and Thoron Daughters. June, p. 201
- Hood, D. B.: Toxicity Studies with Octafluorocyclobutane. October, p. 382
- Hosey, Andrew D: Evaluation of an Aerosol Photometer for Dust Counting and Sizing. December, p. 491
- Houghton, J. A.: Data on Ultraviolet Absorption and Fluorescence Emission. June, p. 219
- Hubbard, D. M.: Determination of Manganese in Air and Biological Material. October, p. 356
- Hueper, W. C.: The Carcinogenic Effects of Single and Repeated Doses of 3,4-Benzpyrene. October, p. 350
- Jacobs, Morris B.: Determination of Mercury in Blood. December, p. 475
- Johnston, Marian S.: Determination of Decaborane by Means of Ferricvanide. August, p. 312
- Jones, Allen R.: A Criterion for Evaluation of Noise Exposures, December, p. 481
- Jones, Herbert H.: Evaluation of an Aerosol Photometer for Dust Counting and Sizing. December, p. 491
- Kathren, Ronald L.: Mycological Contamination of Impinger Dust Samples. June, p. 261
- Katz, R.: Retention and Fate of Iridium-192 in Rats Following Inhalation. October, p. 414
- Kracke, Robert D.: Use of Spider Silk in Aerosol Research. June, p. 263
- Kuhas, Larry J.: Determination of Decaborane and Pentaborane
- by Means of Triphenyltetrazolium Chloride. June, p. 231 Kusnetz, Howard L.: Air Flow Calibration of Direct Reading
- Colorimetric Gas Detecting Devices. August, p. 340 Calibration and Evaluation of Gas Detecting Tubes. October,
- p. 361 LaBelle, C. W.: Retention and Effect of Radioactive Particulates
- in the Lung. June, p. 195

  Lacy, William J.: A Comparison of the Composition of Reactor

  Waste Solutions and Radioactive Fallout. August, p. 334
- Landry, A. S.: The Polarographic Determination of Thallium in an Atmospheric Sample. October, p. 407
- Lanier, Marshall E.: Calibration and Evaluation of Gas Detecting Tubes. October, p. 361
- Lee, George: Data on Ultraviolet Absorption and Fluorescence Emission. June, p. 219
- Levadie, Benjamin: An Application of Gas Chromatography to Analysis of Solvent Vapors in Industrial Air. February, p. 20

- The Determination of Organic Vapors in Air by Chromatography Using a Direct Injection Method. August, p. 322 Leverett, J. K.: An Investigation of Lens Opacity on Personnel
- Operating a Portable Nuclear Reactor. February, p. 84 Lewis, Helen M.: Use of Spider Silk in Aerosol Research. June, p. 263
- Linch, A. L.: Development of a Freeze-out Technique and Constant Sampling Rate for the Portable Uni-Jet Air Sampler. August, p. 325
- Lindeken, C. L.: The Control of Beryllium Hazards. June, p. 245
  Longley, Mars Y.: Pulmonary Deposition of Dust as Affected by Electric Charges on the Body. June, p. 187
- Lundgren, K. D.: A Survey of Results of Investigations on Some Organic Mercury Compounds Used as Fungicides. August, p. 308
- Maga, John A.: Statewide Air Pollution Standards in California. October, p. 430
- Mastromatteo, E.: Acute Inhalation Toxicity of Vinyl Chloride to Laboratory Animals. October, p. 394
- Maynard, Elliott A.: Acute and Sub-acute Toxicity Studies of Thallium Compounds. October, p. 399
- McKelvey, J. W.: Health Aspects of the Commercial Melting of Uranium-Contaminated Ferrous Metal Scrap. April, p. 178 McLaughlin, James E.: Nuclear Safety in Manufacturing Plants. February, p. 59
- McNerney, James M.: The Acute Toxicity of Cyanogen. April, p. 121
- Meadors, O. L.: The Control of Beryllium Hazards. June, p. 245 Mendenhall, R. M.: Measurement of Ketene for Study of Its Toxic Action on Animals. June, p. 211
- Merrill, Jean M.: Determination of Pentaborane in Air by Means of Activated Carbon. February, p. 15
- Determination of Decaborane and Pentaborane by Means of Triphenyltetrazolium Chloride. June, p 231
- Determination of Decaborane by Means of Ferricyanide. August, p. 312
- Miller, Franklin A.: The Determination of n-Butylamine in Air. December, p. 471
- Mitchell, R. I.: A Tracer Technique to Measure Deposition of Stack Emissions. October, p. 343
- Monkman, J. L.: Survey of Lead Hazards in Indoor Firing Ranges. June, p. 256
- Moore, Hezekiah: A Spectrophotometric Method for the Determination of Mercaptans in Air. December, p. 466
- Morrill, E. Elbridge: Tetraethyl Lead Poisoning Incident with Eight Deaths. December, p. 515
- Morse, Robert S.: Urinary Uranium Levels in Non-Exposed Individuals. February, p. 68
- Individuals. February, p. 68

  Musselman, Nelson P.: The Effects of Continuous Exposure of
  Animals to Ethanolamine Vapor. October, p. 374
- Nader, John S.: Constant Flow Regulators for the High-Volume Air Sampler. April, p. 115
  - A Mass Loading and Radioactivity Analyzer for Atmospheric Particulates. August. p. 300
- Nehemias, John V.: The Pre-Operational Environmental Survey for the Enrico Fermi Reactor—1958, April, p. 144
- Oyen, F.: The Toxicity of Bromochloromethane (Methylene Chlorobromide) as Determined on Laboratory Animals.

  August, p. 275
- Pagnotto, Leonard D.: Treatment of Chronic Mercurialism with N-Acetyl-Penicillamine. October, p. 419
- Palm, Betty J.: Determination of Decaborane and Pentaborane by Means of Triphenyltetrazolium Chloride. June, p. 231
- Determination of Decaborane by Means of Ferricyanide. August, p. 312 Payne, William W.: The Carcinogenic Effects of Single and Re-
- Payne, William W.: The Carcinogenic Effects of Single and Repeated Doses of 3,4-Benzpyrene. October, p. 350
- Pecora, Louis J.: Survey of Current Status of Raynaud's Phenomenon of Occupational Origin. February, p. 80
  Pegues, W. Leak: Lead Fume from Welding on Galvanized and
- Zinc-silicate Coated Steels. June, p. 252
- Pfeiffer, Albert: A Simple Mathematical Method for Evaluation

n

61

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of Data from Droplet Counts on Settling and Impaction Slides. December, p. 526

Pihl, Stanley E.: Evaluation of Exposures to Industrial Noise December, p. 486

Rastatter, K. A.: Retention and Effect of Radioactive Particulates in the Lung. June, p. 195

Riedesel, Marvin L.: Laboratory Simulation of a Hot Industrial Job to Find Effective Heat Stress and Resulting Physiologic Strain. February, p. 25

Rinehart, William E.: The Sub-acute and Chronic Toxicity of 1,1-Dimethylhydrazine Vapor. June, p. 207

Studies on the Toxicity of Triethylborane (TEB) October, p. 389

Robbins, M. Chain: Some Health Hazards Associated with the Manufacture of Commercial Jet Aircraft. April, p. 182

Ross, C. R.: Survey of Lead Hazards in Indoor Firing Ranges. June, p. 256

Rowe, V. K.: The Toxicity of Bromochloromethane (Methylene Chlorobromide) as Determined on Laboratory Animals. August, p. 275

Saltzman, Bernard E.: Calibration and Evaluation of Gas Detecting Tubes. October, p. 361

Sawicki, Eugene: Benzo (a) pyrene Content in the Air of American Communities, December, p. 443

Schafer, L. J.: The Concentrations of Oxidant (Ozone) and Nitrogen Dioxide in the Air of Cincinnati, Ohio, December, p. 452

Scherberger, Richard F.: The Determination of n-Butylamine in Air. December, p. 471

Schrenk, H. H.: The Acute Toxicity of Cyanogen. April. p. 121 Schulz, E. J.: A Tracer Technique to Measure Deposition of Stack Emissions, October, p. 343

Scott J. K : Retention and Fate of Iridium-192 in Rats Following Inhalation. October, p. 414

Acute and Sub-acute Toxicity Studies of Thallium Compounds. October, p. 399

Seals, Jack: Determination of Decaborane and Pentaborane by

Means of Triphenyltetrazolium Chloride, June, p. 231 Sheinbaum, Milton: Experiences with Phase Microscopy, Decem-

ber, p. 518 Silis, Vincent: A Generator for Producing Low Concentrations of Vapor in Inhalation Chambers. October, p. 423

Small, B. R.: Factory Heat Waves Can Be Broken. February, p. 32

Stanley, Thomas W.: Benzo(a) pyrene Content of the Air of American Communities. December, p. 443 Starkey, R. H.: Health Aspects of the Commercial Melting of

Uranium-Contaminated Ferrous Metal Scrap. April, p. 178 Steadman, Luville T.: Acute and Sub-acute Toxicity Studies of Thallium Compounds. October, p. 399

Stephan, David G.: Concepts in Fabric Air Filtration. February,

Stephenson, R. W.: Hazards from the Use of Isopolyesters as Maintenance Coatings, December, p. 522

A

Stern, Arthur C.: Survey of Air Pollution Research in Europe. December, p. 459

Swensson, A.: A Survey of Results of Investigations on Some Organic Mercury Compounds Used as Fungicides. August, p. 308

Symons, Noel S.: What is Expected of the Professional Person in Litigation. December, p. 502

Talvitie, N. A.: Separation of Fluoride by Ion Exchange: Application to Urine Analysis, August, p. 287

Tebbens, Bernard D.: Five Years of Continuous Air Monitoring. February, p. 39

Terry, James P.: Developments in the Measurement of Atmospheric Sulfur Dioxide. August, p. 316

Thornton, Joseph H., Jr.: The Inhalation Hazard from Dustproducing Operations in the Repair of Ships with Low-level Radioactive Contamination. February, p. 71

Tom, Baldwin: Mycological Contamination of Impinger Dust Samples, June, p. 261

Torkelson, T. R.: The Toxicity of Bromochloromethane (Methylene Chlorobromide) as Determined on Laboratory Animals. August, p. 275

Udel, Melvin: Survey of Current Status of Raynaud's Phenomenon of Occupational Origin. February, p. 80

Ungar, E. W.: A Tracer Technique to Measure Deposition of Stack Emissions. October, p. 343

Urquiza, Ulises: Determination of Decaborane and Pentaborane by Means of Triphenyltetrazolium Chloride. June, p. 231

Van Wyck, R. W. Protective Clothing Program at the Savannah River Plant. February, p. 55

Vance, Grant H.: Cadmium Exposures in Silver Soldering Operations. April, p. 107

Viles, Frederick J. Jr.: Air Flow Resistance of Flexible Metal Hose. August, p. 296

de Villiers, A. J.: Survey of Lead Hazards in Indoor Firing Ranges. June, p. 256 Walsh, George William: Concepts in Fabric Air Filtration, Feb-

ruary, p. 1 Weeks, Maurice H.: The Effects of Continuous Exposure of

Animals to Ethanolamine Vapor. October, p. 374 Welch, Allan F.: Developments in the Messurement of Atmos-

pheric Sulfur Dioxide, August, p. 316

Welford, George A.: Urinary Uranium Levels in Non-Exposed Individuals. February, p. 68 Whipple, G. Hoyt: The Pre-Operational Environmental Survey

for the Enrico Fermi Reactor-1958. April, p. 144

Williamson, Walter H.: Treatment of Exhaust Gases from Boron Fuels, April, p. 110

Windish, J. P.: Survey of Lead Hazards in Indoor Firing Ranges. June, p. 256

Yamaguchi, Seiya: Determination of Mercury in Blood. December, p. 475

Zinger, B. L.: Retention and Effect of Radioactive Particulates in the Lung, June, p. 195

# Subject Index

-for noise control, 132

spectra of thiols, 466 acetonitrile

-Hygienie Guide, 265

acetylpenicillamine

-for mercurialism, 419

acrylonitrile ultraviolet absorption, 222

addre

of air pollution organizations, 459

aerosol

-counting, 526

-effect of charge, 187 -measurement of, 491

-photometer for, 491

sampling for, 428

study of, 263

-filtration of, 1

air flow

-for gas detectors, 340

-resistance to, 296

В

air pollution —benzpyrene in, 443	animals —electric charge on, 187
-carbon monoxide, 46	anthracene
-chamber design, 423	-ultraviolet absorption, 222
—in Cincinnati, 452	apparatus
—detmng. radioactive dusts, 300	bubbler, 21
-European activities, 459	-bubble meter, 340
-monitoring, 39	—chambers, 211, 239, 423
-seasonal effects, 452 -sources of, 136	—dust counter, 491 —for electric charge, 188
-standards, 430	-gas mixing, 361
-sulfur dioxide in, 316	-hi-vol sampler, 115
alkanethiols	-ketene generator, 211
—detmn. in air, 466	-mass loading analyzer, 300
alkylamines	—freeze-out, 325
—dtmn. in air, 471 allyl benzene	-radiation, 156, 300
—ultraviolet absorption, 222	arsine —detector tube for, 361
aluminum-morin	audiometry
fluorescence of, 228	—use of, 125
-ultraviolet absorption, 222	Australian bubbler, 21
amines	automobiles
—detn. in air, 471	—and air pollution, 46
aminobutane —Hygienic Guide, 532	
analysis	1,2-Benzanthracene
—for alkanethiols, 466	—fluorescence of, 228
alkylamines, 471	benzene
arsine, 361	—detmn., 21
boranes, 15, 231, 312	plant design, 173
butanethiols, 466	—ultraviolet absorption, 222
cadmium, 107 carbon monoxide, 47, 361	—urine sulfates, 511 benzidine
carbon tetrachloride, 21, 322	fluorescence of, 228
chlorine, 361	-ultraviolet absorption, 222
chlorine dioxide, 361	benzoic acid
decaborane, 231, 312	-fluorescence of, 228
ethanethiol, 466	-ultraviolet absorption, 222
ethanolamine, 374	1,12-benzperylene
—by fluorescence, 219 —for fluoride, 287	—fluorescence of, 228 benzpyrene
hexanethiol, 466	—cancer from, 350
hydrogen fluoride, 361	-in city air, 443
ketene 211	-fluorescence of, 228
lead, 253	-ultraviolet absorption, 222
manganese, 356	3,4-benzpyrene-sulfonated
mercaptans, 466	-fluorescence of, 228
mercury, 361, 475 methanethiol, 466	—ultraviolet absorption, 222 beryllium
methylene chloride, 21	—control of hazard, 245
nitric oxide, 87	-detmn. in air, 249
nitrogen dioxide, 87, 361	beryllium ion
organic compounds, 219	—elution with, 287
pentaborane, 15, 231	beryllium-morin
perchloroethylene, 21	—fluorescence of, 228 —ultraviolet absorption, 222
phosgene, 361 radioactive dusts, 300	blood
solvents in air, 20, 322	—carbon monoxide in, 46
stibine, 361	-detmn. manganese in, 356
sulfur dioxide, 316	-detmng. mercury in, 475
thallium, 407	boranes
thoron, 201	—detmn. of, 15, 231, 312
toluene, 22	-triethyl toxicity, 389
trichloroethylene, 21	boron —fuels, 110
uranium, 68	boron-benzoin
xylene, 22	-fluorescence of, 228
aniline	-ultraviolet absorption, 222
—fluorescence of, 228	boron oxide
-ultraviolet absorption, 222	—in air, 110
aniline sulfate	bromochloromethane
-ultraviolet absorption, 222	-toxicity, 275

C

0

colculation -of particle count, 526 calibration -air flow, 340 -of dust counter, 491 -established, 239 -of fluorescent tracer, 343 -of gas detectors, 340, 361 California -air pollution standards, 430 cancer -from benzpyrene, 350 carbon activated, 15 carbon monoxide -detector tube for, 361 -detmn. in air, 47 -detmn. in blood, 47 -standard in air pollution, 430 -from traffic, 46 carbon tetrachloride -detmn. of, 21, 322 -fluorescence of, 228 carbonyl chloride -Hygienic Guide, 530 carcinogen

—dose response, 350
earinomas
—from benzpyrene, 350
chalcedony
—detmn. by phase microscope, 518
chamber
—exposure design, 423
—multi-purpose, 239
charges
—on dust, 187
chipping
—radiation hazard, 71
chlorine
—detector tube for, 361
chlorine dioxide
—detector tube for, 361

—prep. of concentrations, 361 chlorobenzene —ultraviolet absorption, 223 cholanthrene —ultraviolet absorption, 223 chromate ion

-ultraviolet absorption, 223 chromatography -detmn. of solvents, 20, 322 -tubes for, 287 chrysene
—fluorescence of, 228
—ultraviolet absorption, 223
cigarettes
—benzpyrene in smoke, 443
Cincinnati
—air polution in, 452
cleaning
—hands, 162
—jet-fuel tanks, 182
—leaded-fuel tanks, 515
clothing
—and heat load, 25
—radiation protection by, 55

cobalt naphthenate
—as promoter, 522
cold trap
—sampler, 325
color matching
—tests for, 361
colorimetric
—detmn. of boranes, 231
combustion products, of treomposition

--detmin. of boranes, 231 combustion products, of triethylborane, 389 composition --of fallout, 334 --of reactor waste, 334

concentrations

-established, 239

-preparation of known, 88, 361

conservation

-of hearing, 481

contamination

—by fallout, 162

—of impingers, 261

—of river, 156

—by uranium, 178

control
—of benzene hazards, 173
—of beryllium hazard, 245
—of noise, 130
—of radiant heat, 32

of radiation hazards, 59, 178
of radioactive dust, 71
of x-ray units, 436
cooling

—by evaporation, 32
—by ventilation, 32
costs
—of protective clothing, 55

counting
—dust particles, 491, 518
creams
—protective, 522

criterion
—for hearing conservation, 481
critical mass, 61

cresol
—fluorescence of, 228
—ultraviolet absorption, 223

crystal —microphones, 486 cyanogen

-toxicity, 121

daughters
—radon, sampling for, 428
—of thoron, 201
DDT
—fluorescence of, 229

-ultraviolet absorption, 224

D

deaths -from tetraethyl lead, 515 -from vinyl chloride, 394 decaborane -detmn. of, 231, 312 decontamination -of hands, 162 dentist -radiation exposure, 169 deposition -dust in lungs, 187, 195 -of iridium dust, 414 -of stack emissions, 343 dermatitis -from polyesters, 522 detectors -gas tubes, 340 -tubes for gas, 361 determination -(see analysis, also specific substances) diaminodiethylamine -Hygienic Guide, 268 1,2,5,6-dibenzanthracene -ultraviolet absorption, 223 1,2,5,6-dibenzanthracene-sulfonated -fluorescence of, 228 ultraviolet absorption, 223 dibenzpyrene -fluorescence of, 229 -ultraviolet absorption, 223 dibutyl phthalate, -ultraviolet absorption, 224 dicetyl phthalate -ultraviolet absorption, 224 n-dichlorobenzene -ultraviolet absorption, 224 diethylamine -Hygienic Guide, 266 diethyl 1,4-dioxide -Hygienic Guide, 533 diethyldithiocarbamate separation for Mn, 356 diethylene triamine -Hygienic Guide, 268 digestion of blood, 475 dimethylaniline -with polyesters, 522 -ultraviolet absorption, 224 dimethyl benzanthracene cancer from, 350 dimethylhydrazine (UDMH) -toxicity, 207 dimethyl paraphenylenediamine in detmn. of thiols, 466 2,4-dinitrophenol -ultraviolet absorption, 224 2,4-dinitrotoluene -ultraviolet absorption, 224 dioxane -Hygienic Guide, 533 diphenyl -fluorescence of, 229 ultraviolet absorption, 224 diphenyl methane -ultraviolet absorption, 224 dispersal of fluorescent tracer, 343 disposal

-radioactive trash, 151

extraction of mercury, 475

dithizone

of noise, 481 Dowex resins -for ion exchange, 287 droplets -for aerosol study, 263 -calculation of, 526 dust counting -instrument for, 491 -with phase microscope, 518 dust -filtration, 1 -in lungs, 187, 195 -metal hose, 296 -radiation hazard, 71 -radioactive, 300 sampling problem, 261 dyes -uranine as tracer, 343 dynamic -chamber, 239 -microphones, 486 E efficiency of sampling, 428 electric charges on body, 187 elution of fluoride, 287 enclosure -machine operations, 246 -for noise control, 132 Enrico Fermi Reactor, 144 epoxy resins -spraying, 184 ethanolamine -toxicity of, 374 ethanenitrile -Hygienic Guide, 265 ethanethiol -detmn. in air, 466 ethyl benzene -ultraviolet absorption, 224 Europe -air pollution organizations in, 459 excretion of iridium, 414 exhaust gases -boron in, 110 -standards, 430 exposure -to ethanolamine, 374 -noise risk, 481 -to x-ray, 436 exposure chamber -with ketene, 211 extraction -with dithizone, 475 -effects of bromochloromethane, 275 fabric -for filters, 1 fallout -composition of, 334 -synthetic, 162 feces excretion of iridium, 414 ferricyanide -to detmn. decaborane, 312

-for aerosol study, 263 film badges -use of, 169 filter paper sampling with, 428 -in dust counting, 491 -fabric for, 1 -membrane, 518 -for urine sulfate, 511 filtration -air, 1 -theory, 1 regulator for, 115 "flounder" -instrument, 156 fluoranthene -fluorescence of, 229 -ultraviolet absorption, 224 fluorescent -analysis, 219 -tracer technique, 343 fluoride -detmn. of, 287 -in urine, 287 fluorimetric analysis -for uranium, 68 fluorocarbons -toxicity of, 382 fluorography -hazards of, 436 freeze-out sampling, 325 friction -in metal hose, 296 fuels -boron, 110 -tank cleaning, 182, 515 fungicides -mercury poisoning, 308

galvanized metal -welding of, 252 gamma ray -medical exposures, 169 -detecting tubes for, 361 -mixing device, 361 gas chromatography, 20 -for solvents, 322 gas detectors -calibration of, 340 generator -for vapor concentrations, 423 glycerol trinitrate -Hygienic Guide, 99 Griess-Ilosvay reagent, 87

G

H

-conservation criteria, 481 -definitions of, 125 -exposure control, 32 -stress of, 25 hexanethiol -detmn. in air, 466 hi-vol sampler -regulator, 115

-for air flow, 296 humidity and stress, 25 hydraulic fluids, 182 hydrides of metal -Hygienic Guide, 100 hydrocarbons -in air. 443 standard in air pollution, 430 hydrogen fluoride -detector tube for, 361 Hygienic Guides -acetonitrile, 265 -aminobutane, 532 -carbonyl chloride, 530 -diaminodiethylamine, 268 -diethylamine, 266 -diethyl 1,4-dioxide, 533 -diethylene triamine, 268 -dioxane, 533 -ethanenitrile, 265 -glycerol trinitrate, 99 magnesium, 97 -metal hydrides, 100 -methyl cyanide, 265 -molybdenum, 102 -nitroglycerin, 99 -nitropropane, 267 -perchloroethylene, 270 -phosgene, 530 stibine, 529 -tetrachloroethylene, 270 identification

-by fluorescence, 219 -of organic chemicals, 219 -by ultraviolet, 219 impactor -calculations for, 526 impingers -contamination of, 261 index -of heat stress, 25 inhalation -chamber design, 423 -of iridium, 414 -of octafluorocyclobutane, 382 -of vinyl chloride, 394 instruments -automatic dust counters, 491 -for radioactive dusts, 300 iodine -ultraviolet absorption, 224 on exchange -detmn. of fluoride, 287

jet aircraft -hazards in manufacturing, 182 ketene

-for uranium detmn., 68

-inhalation of, 414 isopolyesters

-hazards of, 522

iridium

-detmn. of, 211 -generation, 211 Kruger Mercury Vapor Meter, 475

lead

-detmn. in air, 253

-at firing ranges, 256 -tetraethyl poisoning, 515

-in urine, 256

-in welding fume, 252

lead methyl mercaptide

-in analysis, 466

lead peroxide candle -detmn. of sulfur dioxide, 316

legal

-witness, 330

lens opacity

-from radiation, 84

lindane

-fluorescence of, 229

lithium hydride

-Hygienic Guide, 100

lithium-oxine

-fluorescence of, 229

-ultraviolet absorption, 225

litigation

witness in, 330, 502

lungs

-dust in, 187, 195

-uranium dioxide in, 195

M

magnesium

-Hygienic Guide, 97

magnesium hydride

-Hygienic Guide, 100

manganese

-in biologic material, 356 -detmn. of, 356

mass loading analyzer, 300

melting

-contaminated scrap, 178

membrane filters

-in dust counting, 491, 518

mercaptans -detmn. in air, 466

mercaptide

-of lead, 466

mercury

-detector tube for, 361

-determn. in blood, 475

-organic compounds, 308

-poisoning by, 308

-poisoning cases, 419

metal hose

-for air flow, 296

metal hydrides

-Hygienic Guide, 100

meters

-mercury vapor, 475

methanethiol detmn. in air, 466

methyl cyanide -Hygienic Guide, 265

methylethylketone peroxide

-as promoter, 522

methyl parathion

-ultraviolet absorption, 225

methyl styrene

-ultraviolet absorption, 225 9-methyl anthracene

-fluorescence of, 229

-ultraviolet absorption, 225

methylene chloride

-detmn, of, 21

methylene chlorobromide

-toxicity, 275

methylcholanthrene

-cancer from, 350

20-methylcholanthrene

-fluorescence, 229 -ultraviolet absorption, 225

methoxychlor

-- fluorescence of, 229

-ultraviolet absorption, 225

microphone

-for noise studies, 486

microscopy

-phase, 518

molybdenum

-Hygienic Guide, 102

monitoring

-of air pollution, 39

-beryllium in air, 249

mycological contamination, 261

naphthalene

-ultraviolet absorption, 225

naphthylamine

-fluorescence of, 229

-ultraviolet absorption, 225

nicotine

-ultraviolet absorption, 225 ninhydrin

-reagent for amines, 471

nitric oxide

-detmn. of, 87 nitroaniline

-fluorescence of, 229

-ultraviolet absorption, 225

nitrobenzene

-fluorescence of, 229

-ultraviolet absorption, 225

nitrogen dioxide

-in city air, 452

-detector tube for, 361

-detmn. of, 87

nitroglycerin

-Hygienic Guide, 99 nitrophenol

ultraviolet absorption, 226

nitropropane -Hygienic Guide, 267

nitrotoluene

-ultraviolet absorption, 226

noise

-control of, 130

-dose concept, 481 -and hearing, 125

-limits for, 130

-measurement of, 486

nuclear safety, 59

-lens opacity in, 84

0

octafluorocyclobutane

-toxicity of, 382 octave band analyzer

-use of, 486 oil mists

-fluorescence of, 229

opacities

-of lens, 84

organic mercury poisoning, 308

organic vapors, (see solvents)

0

organizations air pollution, 459 oxidant -in city air, 452 -index of, 430 oxy-acetylene soldering, 107 ozone -in city air, 452 -standard in air pollution, 430 P painting -ероху spray, 184 parathion -ultraviolet absorption, 226 particle size -calculation of, 526 -by phase microscope, 518 -by photometer, 491 particles -count and size, 491, 518, 526 particulates -in air pollution, 39 -in city air, 443 -radioactive, 300 (see also dusts) penicillamine -acetyl, for mercurialism, 419 pentaborane -detmn. of, 15, 231 pentachlorophenol -ultraviolet absorption, 226 perchloroethylene -detmn., 21 -Hygienic Guide, 270 permeability -filter, 1 perylene -fluorescence of, 229 -ultraviolet absorption, 226 petro-chemicals -and health, 173 phase microscopy use of, 518 phenanthrene -ultraviolet absorption, 226 -fluorescence of, 229 -ultraviolet absorption, 226 phenoldisulfonic acid reagent, 88 phosgene detector tube for, 361 -Hygienic Guide, 530 photofluorography -hazards of, 436 photometer -for counting dust, 491 physiologic effects -of heat, 25 polarograph -detmn. of cadmium, 107 -detmn. of thallium, 407 -hazards of, 522 polynuclear hydrocarbons -in air, 443 potassium hydride

-Hygienic Guide, 100

preparation
—of ketene, 211

promoters -in polymers, 522 pulmonary -dust, 187 punch press -noise, 133 pyrene -fluorescence of, 229 -ultraviolet absorption, 226 pyridine -fluorescence of, 229 -ultraviolet absorption, 226 quartz -detmn. by phase microscope, 518 radiation -dosage of, 169 -dose from thoron, 201 dust hazard, 71 -heat, 25 -and lens opacity, 84 -medical exposures, 169 -from photofluorography, 436 -reactor safety, 144 -thoron hazards, 201 -from uranium contamination, 178 radiation safety, 55, 59 radio chemistry, 144 radioactive -decay of thoron, 201 -mass of particulates, 300 -trash disposal, 151 -waste in river, 156 radioactivity -decontamination, 162 -in lungs, 195 -measurement, 144 -in river, 156 radiologist exposure of, 169 radon daughters, sampling for, 428 of urine sulfates, 511 Raynaud's Phenomenon, 80 reactor waste -composition of, 334 reactors, (see also nuclear or radiation) survey of, 144 refractive index -in dust counting, 518 -of membrane filters, 518 regulators -hi-vol sampler, 115 Reissner solution -in detmn. of thiols, 466 resin -for uranium detmn., 68 resins **—ероху**, 184 resistance -to air flow, 296 -filters, 1 retention -dust in lungs, 195 -of iridium dust, 414 reverberation, 130 -noise damage, 481

riveting	stack emissions
—noise, 132	—tracer technique for, 343 standards
rotary tools —injury from, 80	—for air pollutants, 430
	static
S	-chamber, 239
safety	-charge on body, 187
-nuclear, 59	stibine
sampler	-detector tube for, 361
—constant rate, 325	-Hygienic Guide, 529
—freeze-out, 325	stress
-hi-vol regulator, 115	—from heat, 25
sampling	strontium hydride
for butylamine, 471 contamination in, 261	-Hygienic Guide, 100 styrene
—for manganese in air, 356	-hazards of, 522
-for mercaptans, 466	-in phase microscopy, 518
—for radioactivity survey, 144	-ultraviolet absorption, 227
-for radon daughters, 428	sulfate
-for thoron in air, 201	-ratio in urine, 511
sampling	sulfonated naphthalene
sanding	—fluoresence of, 229
-radiation hazard, 71	sulfur dioxide
sarcomas	—detmn. in air, 316
—from benzpyrene, 350	-standard in air pollution, 430
Savannah River Project, 55	survey
scrubbers	—air pollution, 136
—for gases, 110	-of noise hazard, 486 -of reactor, 144
seasons —effects on air pollution, 452	
settling	T
—particle count by, 526	tank
shielding	-hazard in cleaning, 515
-heat control by, 32	target shooting
ship repairing	lead hazard of, 256
-radiation hazard, 71	Tennessee River
silica	-contamination, 156
-detmn. by phase microscope, 518	terphenyl
silver	—fluorescence of, 229
-soldering, 107	-ultraviolet absorption, 227
—particle detmn., 518	testimony —by expert witness, 502
skin	tetrachloroethylene
-effects of bromochloromethane, 275	-Hygienic Guide, 270
Skydrol, 184	tetrachloromercurate
smog	-detmn. of sulfur dioxide, 316
-in Cincinnati, 452	tetraethyl lead
smoke	—cases of poisoning, 515
-tracer technique for, 343	tetralin
sodium hydride	-ultraviolet absorption, 227
Hygienic Guide, 100	tetryl
soldering	—ultraviolet absorption, 227 thallium
—silver, 107 solvent	-detmn. of, 407
—analysis in air, 20	-toxicity of, 399
-detmn. in air, 322	therapy
sound level meter	—for mercurialism, 419
—use of, 486	thoron
sources	-detmn. of, 201
-of air pollution, 136	-hazards of, 201
Southern Research Institute, 491	tissues
spectra	—thallium in, 399
-absorption, 466	toluene
spectrophotometer	-detmn., 22
-detmn. of thiols, 466	—ultraviolet absorption, 227 toluidine
speech hearing level, 126 spider silk	—ultraviolet absorption, 227
-for aerosol study, 263	toxicity
	-of bromochloromethane, 275
spray painting —epoxy resins, 184	-of cyanogen, 121

60

-of ethanolamine, 374

-of fluorocarbons, 382

-of octafluorobutane, 382 -of thallium compounds, 399

-of triethylborane, 389

-of vinyl chloride, 394

-for stack emissions, 343

radioactive, 151

treatment of mercurialism, 419

trials

expert testimony in, 502

trichloroethylene

detmn. of, 21

tridymite

detmn. by phase microscope, 518

triethylborane

-toxicity of, 389

triketohydrindene, (see ninhydrin)

2,4,6-trinitrophenol

ultraviolet absorption, 227

trinitrotoluene

ultraviolet absorption, 227 triphenyltetrazolium chloride

for detmng. borane, 231

tubes

-gas detecting, 361

U

ultraviolet

-absorption data, 219 Uni-Jet Air Sampler, 325

-used as tracer, 343

-contaminated scrap, 178

-safe storage, 59

-in urine, 68, 178 uranium dioxide

-in lungs, 195

urinary sulfate ratio, 175, 511

urine

-detmn. manganese in, 356

-excretion of iridium, 414

-fluoride in, 287

-lead in, 256

-uranium in, 68, 178

urologist

-radiation exposure, 169

velocity

of filtering, 1

ventilation

-for cooling, 32

-metal hose for, 296

-for thoron, 201 vibration

-noise transmission, 132

-physiologic effects, 80

vinyl chloride

-toxicity of, 394

warfarin

-fluorescence of, 229

waste

-from reactor, 334

water

-radioactivity in, 144, 156

welding

-galvanized metal, 252

-lead coated metal, 252

-radiation hazard, 71

-zinc silicate coated metal, 252

witness

-legal, 330

-expert, 502

X

x-ray

exposure of dentist, 169 xylene

detmn. of, 22

xylene

-ultraviolet absorption, 227

Z

zinc oxide

-in welding fume, 252

zinc silicate

-in welding, 252

zirconium-flavonol

-fluorescence of, 229 -ultraviolet absorption, 227



